



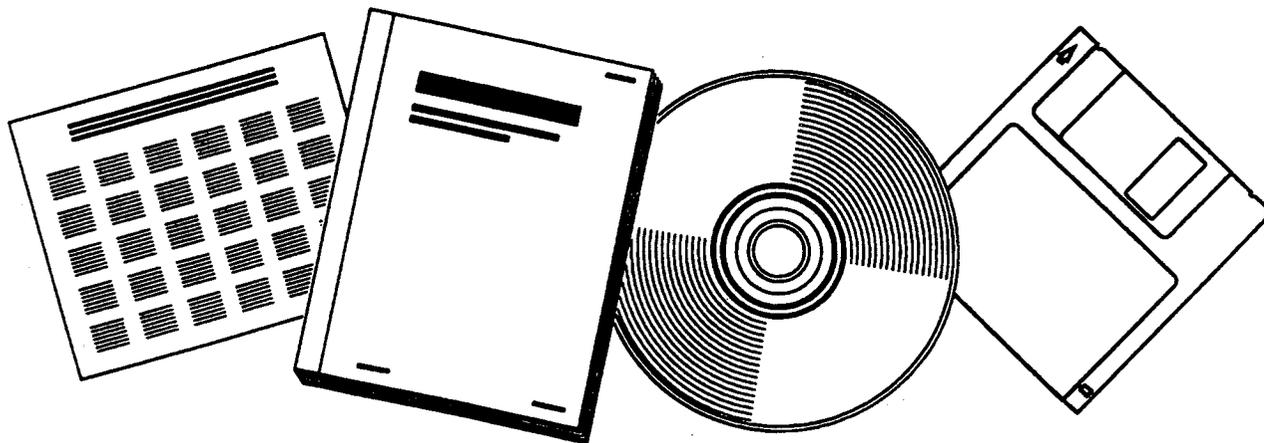
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DEVELOPMENT OF A QUALIFICATION STANDARD FOR ADHESIVES USED IN HYBRID MICROCIRCUITS

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Rockwell International Corporation
Anaheim, CA

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DEVELOPMENT OF A QUALIFICATION STANDARD FOR ADHESIVES
USED IN HYBRID MICROCIRCUITS

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16. ABSTRACT <p>The objective of this program was to develop improved qualification standards and test procedures for adhesives used in microelectronic packaging. The test methods in MSFC-SPEC-592, "Specification for the Selection and Use of Organic Adhesives in Hybrid Microcircuits," were re-evaluated versus industry and government requirements. Four electrically insulative and four electrically conductive adhesives used in the assembly of hybrid microcircuits were selected to evaluate the proposed revised test methods. The intent of this effort was to develop parametric data to establish specification requirements for adhesives used in assembly of hybrid microcircuits for space applications rather than to establish a list of qualified sources for the specification.</p> <p>Based on the results of this study, MSFC-SPEC-592 was revised and rewritten into military specification format. An estimate of the cost to perform qualification testing of an adhesive to the requirements of the revised specification was also prepared.</p>					
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FOREWORD

This report describes work performed from October 1980 to October 1981 under Contract NAS8-33385, Modification No. 3, for the George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Marshall Space Flight Center, Alabama. The technical manager for MSFC was Mr. S. V. Caruso. This report was prepared by Rockwell International, Autonetics Strategic Systems Division, under the direction of Dr. J. J. Licari, Manager of Interconnect Systems Engineering. The principal investigator was Dr. B. L. Weigand. The principal participant in the program was Mrs. C. A. Soykin.

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1.0 INTRODUCTION AND PROGRAM OBJECTIVE

Epoxy adhesives of both the electrically insulative and electrically conductive types are widely used in the assembly of high reliability hybrid microcircuits. The first government specification for procurement of epoxy adhesives for use in hybrid microcircuits was issued for coordination as MSFC Drawing 16A02053 NASA/MSFC in December 1974. The specification was based on work performed by Rockwell International. (1,2) Only a minor number of comments were received from the coordination and the specification was subsequently released as MSFC-SPEC-592, "Specification for the Selection and Use of Organic Adhesives in Hybrid Microcircuits" on May 22, 1978. This specification with minor revisions was also released by the Army Missile Command as MIS 28962 on 29 November 1976 and by the National Security Agency as Specification NSA No. 77-25 on 27 May 1977.

Since implementation of the specification, some problems have been encountered in the qualification of adhesives to its requirements. Areas of concern include outgassing (effects of filler content and effects on devices), bond shear strength (number of specimens, requirement at 150°C, die shear versus lap shear), corrosivity (test method and requirements), volume resistivity (test method), ionic impurities (test method and requirements), electrical stability (test method), and frequency of qualification testing.

The objective of this program was to resolve these problems and to develop improved qualification standards and test procedures for adhesives used in microelectronic packaging.

2.0 PROGRAM SUMMARY

This program consisted of the following five tasks.

2.1 TASK A INDUSTRY REVIEW

Organic adhesives are being used extensively in microelectronic circuits for industry, government, and commercial applications. However, the qualification and selection of specific materials have not been standardized. This has resulted in reliability defects in both short and long term applications. A telephone survey was conducted to determine the state-of-the-art methods used for qualification and control of adhesives and to assess the extent to which MSFC-SPEC-592 was being used in industry.

2.2 TASK B EVALUATION OF TEST METHODS AND REQUIREMENTS

The test methods and requirements in MSFC-SPEC-592 were evaluated versus industry and government requirements using the results from Task A and changes were recommended where required.

2.3 TASK C DEVELOPMENT OF ELECTRICAL STABILITY TEST

Under conditions of time and elevated temperature, certain combinations of conductive adhesives and semiconductor die decrease in conductivity. Then, electrical properties such as V_{CESAT} increase and affect the operation of the device(s). A test method was developed to evaluate the electrical stability of the adhesives when powered at high current densities (to 139.5 amps/cm²) and at a temperature of 150°C. Additional effort would be required to develop a method for evaluating the electrical stability of the large variety of semiconductor die and adhesive combinations taking into consideration the type of die backing, die size, current density, and type of adhesive filler.

2.4 TASK D ADHESIVE EVALUATION

Eight epoxy adhesives, four electrically conductive (two silver filled and two gold filled) and four electrically insulative, were selected to evaluate the test methods and procedures proposed for the revised specification. The intent of this effort was to develop parametric data to be used to establish specification requirements for the adhesives used in assembly of hybrid microcircuits for space applications rather than to establish a list of qualified sources for the specification.

2.5 TASK E SPECIFICATION REVISION

Based on the results of the above tests and the conclusions and recommendations derived therefrom, MSFC-SPEC-592 was revised and rewritten into military specification format.

2.6 TASK F COST DATA FOR MATERIALS TESTING

An estimate was prepared of the cost of material and labor required to perform qualification testing of an adhesive.

3.0 EXPERIMENTAL PROCEDURES AND RESULTS

3.1 TASK A INDUSTRY REVIEW

It was determined that while efforts were underway to qualify adhesives to the specifications, difficulties were being encountered with both the test procedures and test requirements.

Problem areas included:

- (1) Outgassing - Many felt that the metal filled adhesives were favored and that the weight loss should be based on the volume rather than weight of adhesive used.
- (2) Shear strength - The specifications require a minimum of 800 die shear tests for adhesive qualification not including shelf life testing. This was considered excessive. Some preferred lap shear to die shear testing.
- (3) Corrosivity - The simpler aluminized mylar test specified in NAS 77-25A was preferred.
- (4) Volume resistivity - The use of an adhesive strip on a glass slide was preferred over the ASTM D2739 method for electrically conductive adhesives.
- (5) Thermal conductivity - Difficulties were encountered in obtaining data at 150°C. A single test temperature of 121°C was recommended.
- (6) Linear thermal expansion coefficient - Method does not adequately provide for film adhesives.
- (7) Ionic impurities - This test should be required for electrically conductive adhesives also. Difficulties were also encountered in meeting the chloride requirement.
- (8) Electrical stability - An improved test for the effect of time, temperature and current density on the ohmic contact to semiconductor devices is needed.
- (9) Retention of qualification requirements - Most preferred that requalification be required at 24 rather than 12 month intervals.

(10) General comments - The maximum temperature for adhesive cure and use of 150°C was considered by some to be low and does not provide for polyimide adhesives. The use of two specifications, one for the adhesive vendor and one for the microcircuit manufacturer was also recommended.

The survey revealed that General Dynamics is using MIS-28962 for the qualification of adhesives. Martin Marietta is using both MIS-28962 and NSA No. 77-25 and Beckman is using NSA 77-25. No companies were qualifying to MSFC-SPEC-592; however, it should be noted that both the NSA and Army specifications are based on the MSFC specification.

3.2 TASK B EVALUATION OF TEST METHODS AND REQUIREMENTS

Based on a review of MSFC-SPEC-592 and the problem areas discussed above, the following changes in test methods and requirements were selected for evaluation:

3.2.1 INFRARED SPECTRUM

This method was changed to permit solvent extraction of the resin from the filler. No changes in requirements were proposed.

3.2.2 OUTGASSING

It was proposed to make the manufacturer of the adhesive responsible for determining the thermal stability of the adhesive at 250°C using thermogravimetric analysis (TGA). The weight loss at 250°C must be \leq 0.3 wt. percent. The microcircuit manufacturer must insure that his cure, vacuum bake, and sealing process will provide a package ambient meeting the requirements of MIL-STD-883, Method 1018. The short and long term weight loss requirements were deleted.

3.2.3 HARDNESS

It was proposed to delete this test which is of little

benefit as a qualification test and is of concern only in some microwave applications.

3.2.4 BOND SHEAR STRENGTH

The bond shear strength test was revised to reduce the number of shear tests from approximately 800 to a total of 100 per adhesive. Die shear rather than lap shear testing was retained since the former test represents the actual use configuration. Since the adhesion of large capacitors is very dependent on the bonding process (i.e. amount of fillet, total area bonded, use of electrically insulative adhesive for the total bond or for bonding the center of the capacitor), it was proposed to require the microcircuit manufacturer to perform this test. Performance of the semiconductor and substrate die shear tests will be required of the adhesive manufacturer and made optional for the microcircuit manufacturer.

3.2.5 CORROSIVITY

The aluminized mylar corrosivity test specified in NSA No. 77-25A was evaluated in place of the more complex voltage bias-humidity test now specified in MSFC-SPEC-592.

3.2.6 VOLUME RESISTIVITY

The volume resistivity test method specified in NSA No. 77-25A was evaluated in place of the ASTM D2739 method now specified in MSFC-SPEC-592.

3.2.7 THERMAL CONDUCTIVITY

Thermal conductivity measurements are presently required "at approximately 70°C, 120°C and 150°C". It was proposed that this requirement be revised to measure at $121 \pm 5^\circ\text{C}$ only. This temperature approximates the maximum junction temperature in most semiconductor devices and would be the value most commonly

required in heat conduction studies. Thermal conductivity normally decreases as the temperature increases so the use of a value obtained at 121°C would provide a conservative value for conduction calculations at lower temperatures.

3.2.8 LINEAR THERMAL EXPANSION COEFFICIENT

Due to the difficulties involved in preparing specimens of the film adhesives, it was proposed to limit this test to paste adhesives only. For the film adhesives the expansion in the X and Y directions would be comparable to the glass alone and this value could be used in any calculations necessary. The expansion of the film adhesives in the Z direction is not critical to device or substrate adhesion or cracking in most applications.

3.2.9 IONIC IMPURITIES

This test was made a requirement for both electrically conductive and electrically insulative adhesives. Requirements for resistivity, pH, chloride, sodium, and potassium were based on the results of the tests performed in Task D (Paragraph 3.4.9).

3.2.10 DIELECTRIC CONSTANT AND DISSIPATION FACTOR

The dielectric constant and dissipation factor of the electrically insulative adhesive were determined in accordance with ASTM D150 at frequencies of 1KHz and 1 MHz at 25C.

3.3 TASK C DEVELOPMENT OF ELECTRICAL STABILITY TEST

In order to determine the electrical stability of the electrically conductive adhesives, volume resistivity versus temperature and current density measurements were performed over the temperature range of -65 to 150°C using current densities of 7.75 to 186 amps/cm² (50 amps/in² to 1200 amps/in²). The results are shown in Table 1. The results are an average of three specimens/adhesive. The specimens and measurement procedure were identical to those described in paragraph 3.4.6.1 for measuring volume resistivity.

Both silver filled adhesives, Epo-Tek H20E and Ablebond 36-2, showed no change in volume resistivity with increasing current density up to 186 amps/cm² (1200 amps/in²). The gold filled adhesives however, showed evidence of resistive heating at current densities of 46.5 amps/cm (300 amps/in²) and greater for Epo-Tek H44 and 62 amps/cm² (400 amps/in²) and greater for Ablebond 58-6. Based on the TCR values, the calculated temperature increases for the gold filled adhesives with 186.0 amps/cm² (1200 amps/in²) applied are given in Table 2. The temperature coefficient of resistivity was calculated using the equation:

$$\text{TCR}(\text{ppm}/^\circ\text{C}) = \frac{(R_2 - R_1) \times 10^6}{R_1 (T_2 - T_1)}$$

were R_2 = resistivity at the higher temperature T_2

R_1 = resistivity at the lower temperature T_1

The temperature rise was calculated using the TCR values obtained at current density 7.75 amps/cm² and the difference between the resistivity at 186 and 7.75 amps/cm² as the $R_2 - R_1$ value. These temperature increases could seriously impact the validity of any thermal analysis performed on a microcircuit if they are not taken into consideration when using gold filled adhesives. While a current density of 1200 amps/in² may be considered to be high, small signal transistors 0.02 x 0.02 inches square may have currents up

TABLE 1 VOLUME RESISTIVITY (MILLIOHM-CM) vs TEMPERATURE AND CURRENT DENSITY

Adhesive	Temp (°C)	Current Density (A/cm ²)													
		7.750	15.50	31.00	46.50	62.00	77.50	93.00	108.50	124.00	139.50	155.00	170.50	186.00	
Epo-Tek H20E	-65	0.147	←											→	0.147
	0	0.176	←											→	0.174
	25	0.185	←											→	0.185
	50	0.196	←											→	0.196
	100	0.218	←											→	0.218
	150	0.235	←											→	0.235
Epo-Tek H44	-65	0.652	↔		→	0.652	0.653	0.654	0.654	0.655	0.657	0.659	0.661	0.663	
	0	0.791	←		→	0.791	0.792	0.792	0.793	0.794	0.797	0.798	0.801	0.804	
	25	0.844	↔	0.844	0.845	0.846	0.847	↔	0.847	0.849	0.851	0.853	0.856	0.860	
	50	0.899	↔	0.899	0.900	0.900	0.901	↔	0.901	0.904	0.905	0.907	0.910	0.913	
	100	1.010	←					→	1.010	1.020	↔	1.020	1.030	1.030	
	150	1.120	←						→	1.120	1.130	↔	1.130	1.140	
Ablebond 36-2	-65	0.132	←											→	0.132
	0	0.157	←											→	0.157
	25	0.167	←											→	0.167
	50	0.177	←											→	0.177
	100	0.197	←											→	0.197
	150	0.214	←											→	0.214
Ablebond 58-6	-65	0.822	0.821	↔	0.821	0.822	0.823	0.823	0.824	0.826	0.828	0.831	0.834	0.836	
	0	0.989	↔	↔	0.989	0.990	0.993	0.993	0.994	0.995	0.998	1.000	1.000	1.010	
	25	1.050	↔	↔	1.050	1.060	←			→	1.060	1.070	1.070	1.080	
	50	1.130	←						→	1.130	1.140	↔	1.140	1.150	
	100	1.260	←					→	1.260	1.270	↔	1.270	1.280	1.280	
	150	1.460	←				→	1.460	1.470	1.470	1.480	1.490	1.500	1.510	1.520

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TABLE 2 CALCULATED RESISTIVE HEATING OF GOLD FILLED ADHESIVES
WITH CURRENT DENSITY OF 186 A/cm² (1200 A/in²) APPLIED

<u>Substrate Temp. (°C)</u>	<u>Epo Tek H44</u>		<u>Ablebond 58-6</u>	
	<u>Calc. TCR</u>	<u>Calc. Temp. Rise (°C)</u>	<u>Calc. TCR</u>	<u>Calc. Temp. Rise (°C)</u>
-65	3280	5.1	3126	5.4
0	2680	6.1	2467	8.6
25	2607	7.3	3048	9.4
50	2469	6.3	2301	7.7
100	2178	9.1	3175	5.0
150	1800*	9.9	3000*	13.7

* Estimated Value

to 0.5 amps applied yielding a current density of 1250 amps/in².

The effects of increasing current density on the volume resistivity found in this study do not agree with the results reported by Dr. Jerry E. Sergent (3). Sergent found current densities greater than 15.92 A/cm² (102.7 A/in²) caused a decrease in the volume resistivity of all 15 electrically conductive adhesives which he evaluated including the four adhesives studied in this program. The specimen configuration used by Sergent differed in that the materials were cast in a Rexolite mold with a line width and thickness of 8.0×10^{-2} cm and 0.157 cm, respectively, giving a cross-sectional area of 1.26×10^{-2} cm². The line width, thickness, and cross-sectional area used in this study were approximately 0.25 cm, 7.62×10^{-3} cm, and 1.94×10^{-3} cm², respectively. A new test specimen 0.127 x 0.051 cm in width and thickness, respectively, was prepared on a glass slide using Ablebond 36-2. Our previous measurements used a constant voltage of 5 volts. The voltage used by Sergent was not specified. Current density measurements were performed at 25°C on the new specimen using voltages of 0.1, 0.4, 1, 2, 4, 8, and 10 with current densities to 591 amps/in². No changes in the volume resistivity with voltage or current were noted. Based on the above, no reason for the differences in results can be given.

Little is known about the current carrying capability of the conductive adhesives over either the long or short term. The data reported above indicates that the silver filled adhesives can tolerate current densities of at least 186 amps/cm² for short periods of time. Resistive heating of the gold-filled adhesives could make them less reliable than the silver filled at high-current densities.

It was planned to use the current density at which a 5 percent decrease in volume resistivity occurred as a guide in establishing a current density limit for the adhesives. Since

no decrease occurred, the following method was selected for evaluating the electrical stability of the conductive adhesives at 150°C and current densities of 0, 54.3, 93 and 139.5 amps/cm² (0, 350, 600 and 900 amps/inch²).

Thermal dissipation would be a major problem in continuous burn-in of transistors or diodes at high current densities and temperatures. The contact resistance at the interface between the transistors or diodes and adhesives has also shown a dependence on the type of adhesive (i.g., gold or silver filled), the type of backing on the device and the size of the device, as well as the current density applied. (3,4,5) Resolution of all of these problems is beyond the scope of this program. Kovar tabs (gold plated) were therefore selected to evaluate the electrical stability of the adhesives at high temperature and current densities independent of the effects of the interface interactions.

The test specimens consisted of gold plated Kovar tabs 0.0635 x 0.0635 cm (0.025 x 0.025 inch) bonded to thin film gold conductors on alumina substrates using the adhesive to be evaluated. The tabs and conductors were interconnected with ultrasonic bonded gold wires (0.0381 mm) to form a series of nine interconnected tabs as shown in Figure 1. Two series of tabs, each series bonded with a different adhesive were used per substrate (3.81 x 1.91 cm) and three substrates were used for each current density evaluated. The adhesive fillet was removed from around the tabs to control the bond area to 0.00403 cm² per tab. Each series of tabs was individually biased at 5.3 ± 0.5 volts using resistors to control the current densities. Contact to the thin film gold conductors was made using spring loaded contacts in the fixture shown in Figure 2.

The test fixtures were placed under bias in an oven at 150°C containing a nitrogen ambient. Resistance measurements were made initially and at the intervals shown in

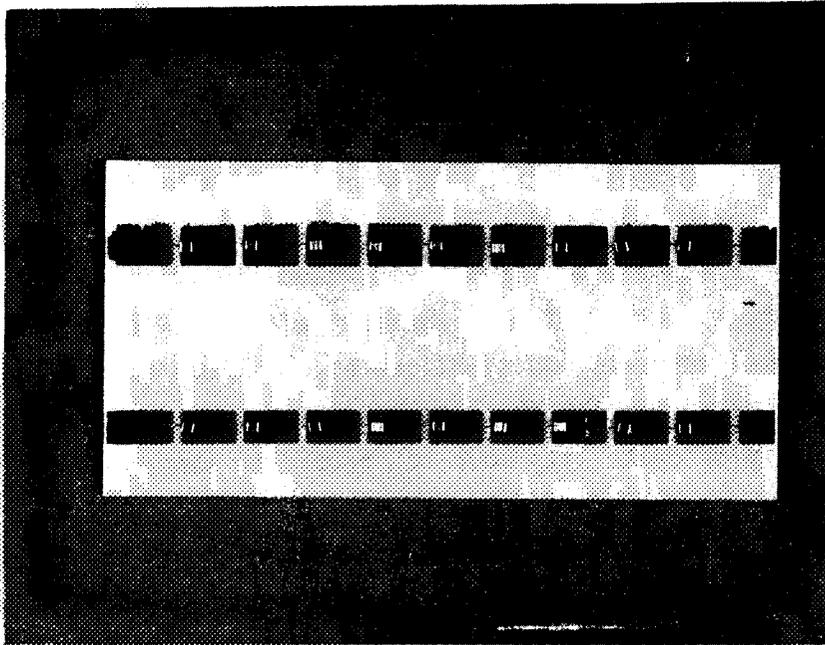


Figure 1. Electrical Stability Test Specimen

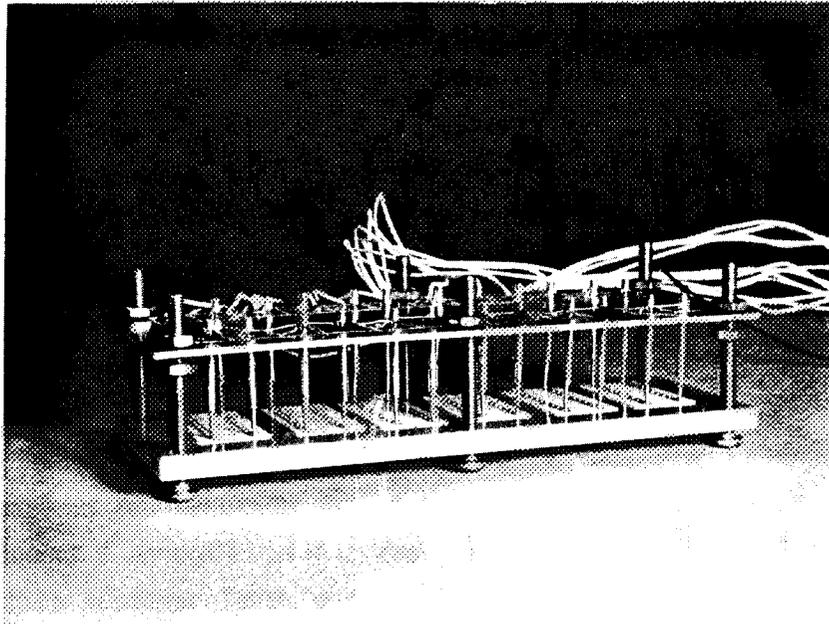


Figure 2. Electrical Stability Test Fixture

Tables 3 through 6. All measurements were made at 25°C using four point probes (Keithley Model 191 Digital Multimeter).

All of the adhesives showed little or no change in the measured resistances after 1000 hours at 150°C and current densities of 0, 54.3, 93, and 139.5 amps/cm². Some instability in the resistance measurements was noted for all four adhesives at a current density of 54.3 amps/cm² after 260 hours (Table 4) but all measurements had stabilized at 550 hours and thereafter. These results indicate that the adhesives have excellent current carrying capability when not bonded to semiconductor devices.

Resistance measurements were also made from the top of the Kovar tabs to the thin film conductors at the conclusion of the test using the substrates which had been powered at 139.5 amps/cm². The resistances measured by the four point probe averaged 0.024 ohms for the two gold filled adhesives and 0.019 ohms for the two silver filled adhesives. Assuming the primary resistance is due to the adhesive and calculating for the volume resistivity gave the results shown in Table 7. Comparison of these resistivity values with those in Table 2 shows the resistivities of the silver filled adhesives to be approximately 500 times higher while those of the gold filled adhesives are approximately 100 times higher than those previously measured using adhesive strips. Mason's⁽⁴⁾ results (Table 7) were 7,800 times higher using an unidentified silver filled adhesive for attachment of a semiconductor. The contact resistance at the adhesive interface is therefore much greater than that indicated by the measurements performed using the adhesive strip on a glass slide. Figures 3 through 6 are SEM photographs (2048X) showing the metal particle distribution in the adhesives. The silver platelets (Figures 3 and 4) are parallel to and show fewer contacts at the bond interface than the more spherical gold particles (Figures 5 and 6). This may explain the higher contact resistance for the silver filled adhesives.

TABLE 3 RESULTS OF ELECTRICAL STABILITY TESTS
AT 150°C AND ZERO CURRENT DENSITY

Adhesive Specimen	Resistance (ohms) ^a					
	Hours at 150°C					
	0	260	400	550	715	1000
Ablebond 36-2 (1)	0.926	0.952	0.960	0.519	0.894	0.960
Ablebond 36-2 (2)	0.837	0.844	0.845	0.853	0.848	0.851
Ablebond 36-2 (3)	<u>0.922</u>	<u>0.896</u>	<u>0.894</u>	<u>0.510</u>	<u>0.960</u>	<u>0.960</u>
Average	0.895	0.897	0.900	0.627	0.901	0.924
Epo-Tek H20E (1)	0.885	0.874	0.874	0.869	0.875	0.873
Epo-Tek H20E (2)	0.912	0.888	0.887	0.882	0.890	0.888
Epo-Tek H20E (3)	<u>0.866</u>	<u>0.851</u>	<u>0.840</u>	<u>0.845</u>	<u>0.856</u>	<u>0.853</u>
Average	0.888	0.871	0.867	0.865	0.874	0.871
Ablebond 58-6 (4)	0.950	0.919	0.918	0.906	0.839	0.913
Ablebond 58-6 (5)	1.056	1.054	1.059	1.058	1.065	1.063
Ablebond 58-6 (6)	<u>1.075</u>	<u>1.056</u>	<u>1.060</u>	<u>1.054</u>	<u>1.060</u>	<u>1.056</u>
Average	1.027	1.010	1.012	1.006	0.988	1.011
Epo-Tek H44 (4)	1.118	1.102	0.598	1.103	0.559	1.109
Epo-Tek H44 (5)	1.029	1.010	1.009	1.008	1.023	1.020
Epo-Tek H44 (6)	<u>0.951</u>	<u>0.513</u>	<u>0.946</u>	<u>0.390</u>	<u>0.518</u>	<u>0.495</u>
Average	1.033	0.875	0.851	0.834	0.700	0.875

^aCombined resistance of 9 gold plated Kovar tabs (0.0635 x 0.0635 cm) adhesively bonded in series to thin film gold conductors and wire bonded with 0.038 mm gold wire.

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**TABLE 4 RESULTS OF ELECTRICAL STABILITY TESTS
AT 150°C AND CURRENT DENSITY OF 54.3 AMPS/CM²
(350 AMPS/INCH²)**

Adhesive Specimen	Resistance (ohms) ^a					
	Hours at 150°C					
	0	260	400	550	715	1000
Ablebond 36-2 (7)	0.580	1.0 f ^b	1.0 f	1.012	1.018	1.014
Ablebond 36-2 (8)	0.920	0.926	0.931	0.920	0.932	0.931
Ablebond 36-2 (9)	<u>0.581</u>	<u>1.043</u>	<u>1.046</u>	<u>1.050</u>	<u>1.091</u>	<u>1.067</u>
Average	0.694	0.990	0.992	0.994	1.014	1.004
Epo-Tek H20E (7)	0.983	0.960	0.948	0.955	0.956	0.947
Epo-Tek H20E (8)	0.888	0.850	0.868	0.869	0.871	0.876
Epo-Tek H20E (9)	<u>0.978</u>	<u>0.960</u> f	<u>0.966</u>	<u>0.956</u>	<u>0.999</u>	<u>0.927</u>
Average	0.950	0.923	0.927	0.927	0.925	0.917
Ablebond 58-6 (10)	1.090	1.30 f	1.064	1.043	1.053	1.041
Ablebond 58-6 (11)	1.062	1.0 f	1.074	1.060	1.060	1.057
Ablebond 58-6 (12)	<u>1.054</u>	<u>0.987</u>	<u>1.020</u>	<u>1.017</u>	<u>1.023</u>	<u>1.015</u>
Average	1.069	1.096	1.053	1.040	1.045	1.049
Epo-Tek H44 (10)	0.996	0.963 f	0.971	0.970	0.975	0.972
Epo-Tek H44 (11)	1.001	0.950	0.986	0.970	0.975	0.978
Epo-Tek H44 (12)	<u>0.991</u>	<u>0.910</u>	<u>0.971</u>	<u>0.962</u>	<u>0.962</u>	<u>0.965</u>
Average	0.996	0.941	0.976	0.967	0.971	0.972

^a Combined resistance of 9 gold plated Kovar tabs (0.0635 x 0.0635 cm) adhesively bonded in series to thin film gold conductors and wire bonded with 0.038 mm gold wire.

^b_f = fluctuating resistance.

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TABLE 5 RESULTS OF ELECTRICAL STABILITY TESTS
AT 150°C AND CURRENT DENSITY OF 93 AMPS/CM²
(600 AMPS/INCH²)

Adhesive Specimen	Resistance (ohms) ^a					
	Hours at 150°C					
	0	260	400	550	715	1000
Ablebond 36-2 (13)	0.952	0.930	0.927	0.927	0.927	0.926
Ablebond 36-2 (14)	0.936	0.920	0.918	0.920	0.915	0.921
Ablebond 36-2 (15)	<u>0.945</u>	<u>0.942</u>	<u>0.948</u>	<u>0.935</u>	<u>0.939</u>	<u>0.938</u>
Average	0.944	0.931	0.931	0.927	0.927	0.928
Epo-Tek H20E (13)	0.889	0.878	0.875	0.876	0.879	0.881
Epo-Tek H20E (14)	0.882	0.922	0.862	0.853	0.857	0.856
Epo-Tek H20E (15)	<u>0.873</u>	<u>0.856</u>	<u>0.862</u>	<u>0.845</u>	<u>0.849</u>	<u>0.848</u>
Average	0.881	0.885	0.866	0.858	0.862	0.862
Ablebond 58-6 (16)	0.974	0.934	0.933	0.930	0.927	0.921
Ablebond 58-6 (17)	1.064	1.035	1.040	1.030	1.037	1.034
Ablebond 58-6 (18)	<u>1.055</u>	<u>1.034</u>	<u>1.037</u>	<u>1.029</u>	<u>1.030</u>	<u>1.032</u>
Average	1.031	1.001	1.003	0.996	0.998	0.996
Epo-Tek H44 (16)	1.063	1.047	1.049	1.031	1.043	1.048
Epo-Tek H44 (17)	0.951	0.930	0.933	0.930	0.932	0.922
Epo-Tek H44 (18)	<u>0.992</u>	<u>0.960</u>	<u>0.962</u>	<u>0.959</u>	<u>0.956</u>	<u>0.963</u>
Average	1.002	0.979	0.981	0.973	0.977	0.978

^a Combined resistance of 9 gold plated Kovar tabs (0.0635 x 0.0635 cm) adhesively bonded in series to thin film gold conductors and wire bonded with 0.038 mm gold wire.

TABLE 6 RESULTS OF ELECTRICAL STABILITY TESTS
AT 150°C AND CURRENT DENSITY OF 139.5 AMPS/CM²
(900 AMPS/INCH²)

Adhesive Specimen	Resistance (ohms) ^a					
	Hours at 150°C					
	0	260	400	550		
Ablebond 36-2 (19)	0.944	0.956	0.945	0.940	0.956	0.926
Ablebond 36-2 (20)	1.059	1.010	1.069	1.080	1.065	1.049
Ablebond 36-2 (21)	<u>0.962</u>	<u>0.963</u>	<u>0.999</u>	<u>0.953</u>	<u>0.885</u>	<u>0.933</u>
Average	0.988	0.976	1.004	0.991	0.969	0.969
Epo-Tek H20E (19)	0.874	0.851	0.853	0.852	0.859	0.836
Epo-Tek H20E (20)	1.009	0.993	1.003	0.968	1.010	0.994
Epo-Tek H20E (21)	<u>0.910</u>	<u>0.886</u>	<u>0.890</u>	<u>0.887</u>	<u>0.896</u>	<u>0.842</u>
Average	0.931	0.910	0.915	0.902	0.922	0.891
Ablebond 58-6 (22)	0.994	0.958	0.963	0.954	0.956	0.933
Ablebond 58-6 (23)	1.051	1.024	1.035	1.025	1.028	1.015
Ablebond 58-6 (24)	<u>0.972</u>	<u>0.958</u>	<u>0.957</u>	<u>0.954</u>	<u>0.960</u>	<u>0.871</u>
Average	1.006	0.980	0.985	0.978	0.981	0.940
Epo-Tek H44 (22)	1.082	1.056	1.058	1.064	1.058	1.056
Epo-Tek H44 (23)	0.978	0.960	0.965	0.962	0.969	0.961
Epo-Tek H44 (24)	<u>1.054</u>	<u>1.050</u>	<u>1.058</u>	<u>1.040</u>	<u>1.046</u>	<u>1.028</u>
Average	1.038	1.022	1.027	1.022	1.024	1.015

^a Combined resistance of 9 gold plated Kovar tabs (0.0635 x 0.0635 cm) adhesively bonded in series to thin film gold conductors and wire bonded with 0.038 mm gold wire.

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ADHESIVE	MEASURED RESISTANCE (ohms)	MEASURED BOND THICKNESS (cm)	BOND AREA (cm ²)	CALCULATED VOLUME RESISTIVITY (ohm-cm)
Abtebond 36-2	0.019	0.001	0.00403	0.077
Epo-Tek H20E	0.019	0.001	0.00403	0.077
Abtebond 58-6	0.024	0.001	0.00403	0.097
Epo-Tek H44	0.024	0.001	0.00403	0.097
Mason's Data (4)	0.12	0.000254	0.00165	0.780

TABLE 7. VOLUME RESISTIVITY CALCULATED USING KOVAR TABS
BONDED TO THIN FILM GOLD CONDUCTORS



Figure 3. SEM Photograph of Ablebond 36-2
Attached Gold-Plated Kovar Tab

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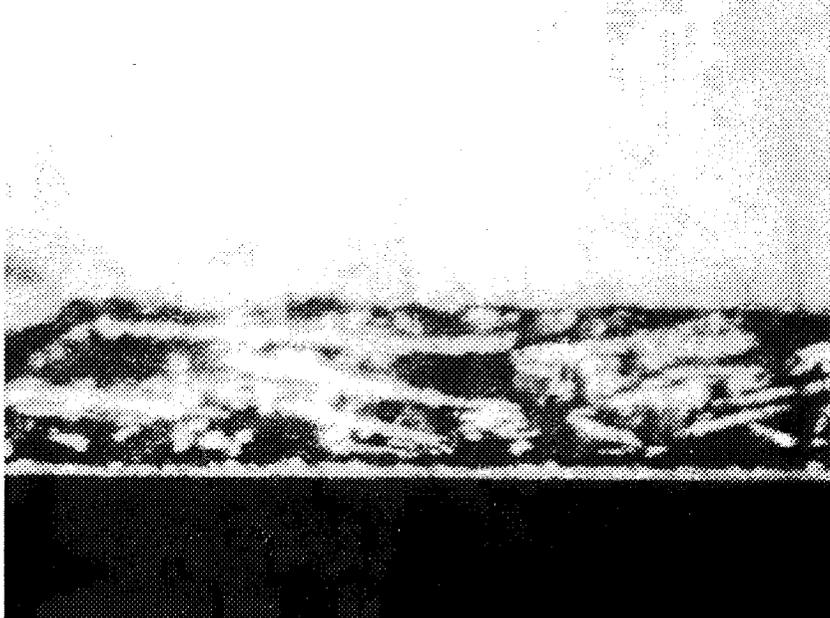
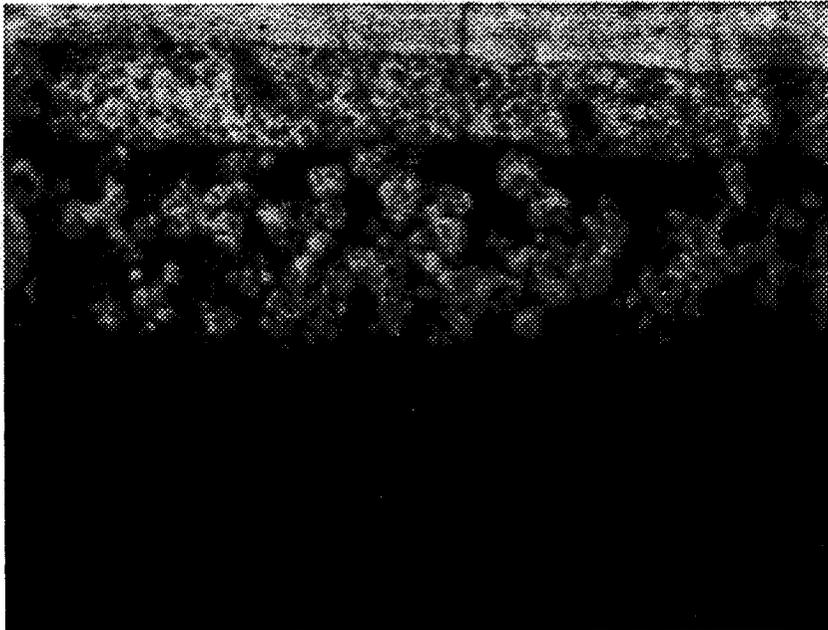


Figure 4. SEM Photograph of Epo-Tek H20E
Attached Gold-Plated Kovar Tab



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Figure 5. SEM Photograph of Ablebond 58-6
Attached Gold-Plated Kovar Tab

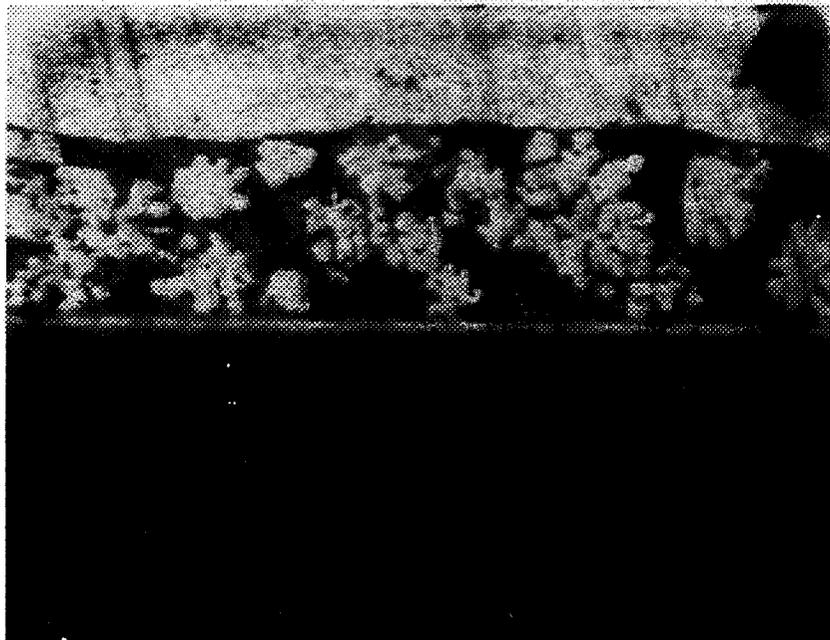


Figure 6. SEM Photograph of Epo-Tek H44
Attached Gold-Plated Kovar Tab

Mason (4) attributed the nonohmic contact obtained between semiconductor dice which did not have properly sintered gold backings and silver-filled epoxy to the metal-semiconductor rectifying junction formed at the semiconductor-junction interface. He also found that silicon transistors greater than 0.08 inch square, when attached with silver-filled epoxy, exhibit an initial increase in contact resistance when exposed to high temperature and then decline to a value near the initial value. He attributed these changes to a pressure buildup of trapped gases which gradually diffuse to the atmosphere. Li (5) found a linear relationship between the die elevation slope and the long term weight loss characteristics of a silver-filled and a gold-filled epoxy. He used this data to define a maximum die size for a given epoxy. Additional studies are required to verify this relationship and to determine the effect of step cures, cure duration and temperature, and vacuum bake, either during or following cure, on the stability of the ohmic contact to semiconductor devices.

3.4 TASK D ADHESIVE EVALUATION

3.4.1 SELECTION OF ADHESIVES

Eight adhesives currently in use for assembly of micro-circuits were selected for evaluation of the test methods and procedures proposed for the revised specification in paragraph 3.2. The results of these tests were used to establish parametric requirements for the specification. A list of the selected adhesives is given in Table 8. Four of the adhesives are electrically conductive (two silver filled and two gold filled) and four are electrically insulative (including one film adhesive).

TABLE 8. ADHESIVES SELECTED FOR EVALUATION OF QUALIFICATION TESTS

<u>Adhesive</u>	<u>Type</u>	<u>Vendor</u>
Epo-Tek H20E	2 Component Silver Filled	Epoxy Technology
Epo-Tek H44	Single Component Gold Filled	Epoxy Technology
Epo-Tek H70E	2 Component, Nonconductive Filler	Epoxy Technology
Epo-Tek H77	2 Component, Nonconductive Filler	Epoxy Technology
Ablebond 36-2	Single Component Silver Filled	Ablestik Laboratories
Ablebond 58-6	Single Component Gold Filled	Ablestik Laboratories
Ablefilm 550	Glass Supported Film, Non-conductive	Ablestik Laboratories
Ablebond 41-1	Single Component Calcium Carbonate Filled Nonconductive	Ablestik Laboratories

3.4.2 INFRARED ANALYSIS

The infrared spectra of the adhesives are shown in Figures 7 through 17. Comparison of the spectra of Ablebond 36-2, Ablebond 58-6, and Ablebond 41-1, Figures 7 through 9 respectively, shows the three adhesives contain identical epoxy resins and curing agents and differ only in the fillers used. The epoxy resin is reported (Ablestik Laboratories) to be resorcinol diglycidyl ether while the curing agent is reported to be an aromatic substituted urea. Epo-Tek H20E and H70E also contain identical epoxy resins, Figures 10 and 11, and curing agents, Figures 12 and 13. The resin and curing agent were not identified. The spectrum of Ablefilm 550 reported (Ablestik Laboratories) to consist of a mixture of a nitrile-modified epoxy and dicyandiamide curing agent is shown in Figure 14. The spectra of Epo-Tek H44 and Epo-Tek H77, Parts A and B, are shown in Figures 15 through 17, respectively. The epoxy resins and curing agents used for these adhesives were not identified.

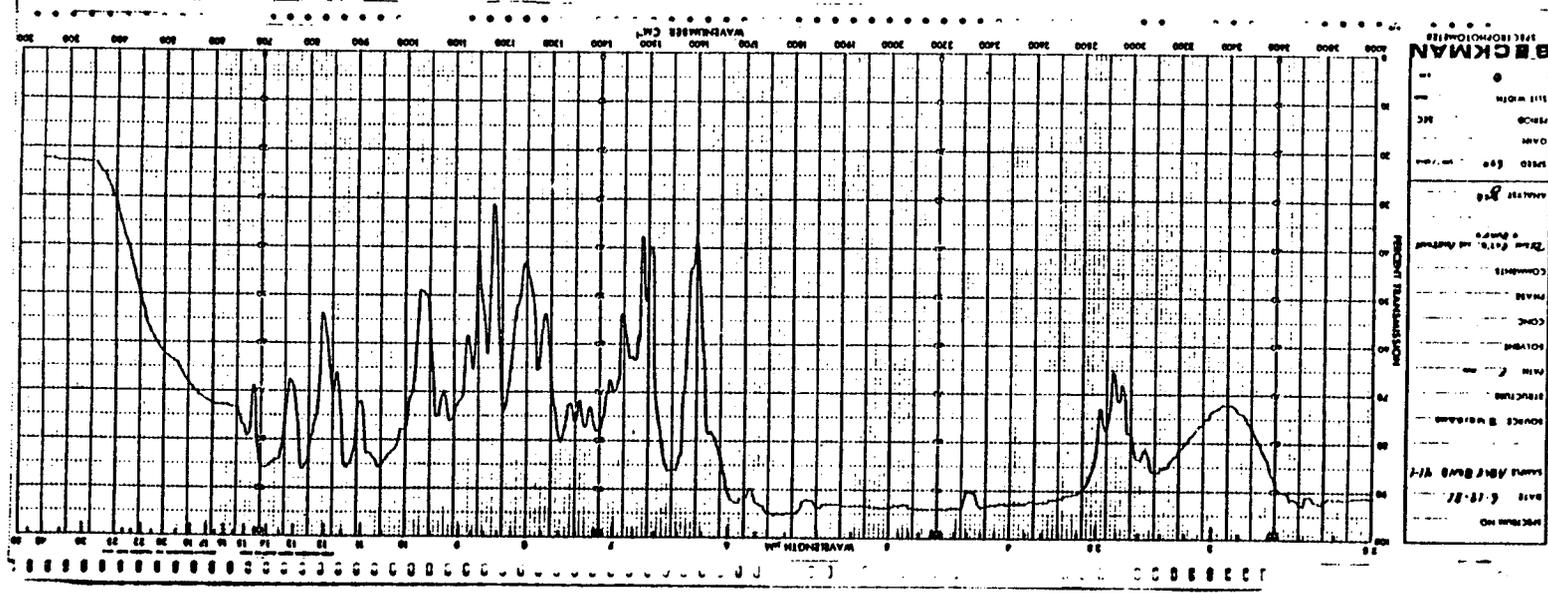
The infrared spectra were obtained by separation of the fillers from the resins and/or curing agents using acetone as the solvent followed by evaporation of the acetone on the sodium chloride window prior to analysis.

3.4.3 OUTGASSING ANALYSIS

The thermal stability of the adhesives was determined as a function of temperature up to 450°C in nitrogen. The results are shown in Figures 18 through 26. The NASA specification requirement is a weight loss ≤ 0.3 wt % at 250°C. The measured weight losses, manufacturers' recommended, and actual cure schedules used are shown in Table 9. It can be seen that the cure schedules would have to be extended beyond those used for the Epo-Tek H20E, Epo-Tek H70E, Ablebond 41-1 and Ablefilm 550 in order to meet the weight loss requirement as it now stands.

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Figure 9. Infrared Spectrum of Ablebond 41-1



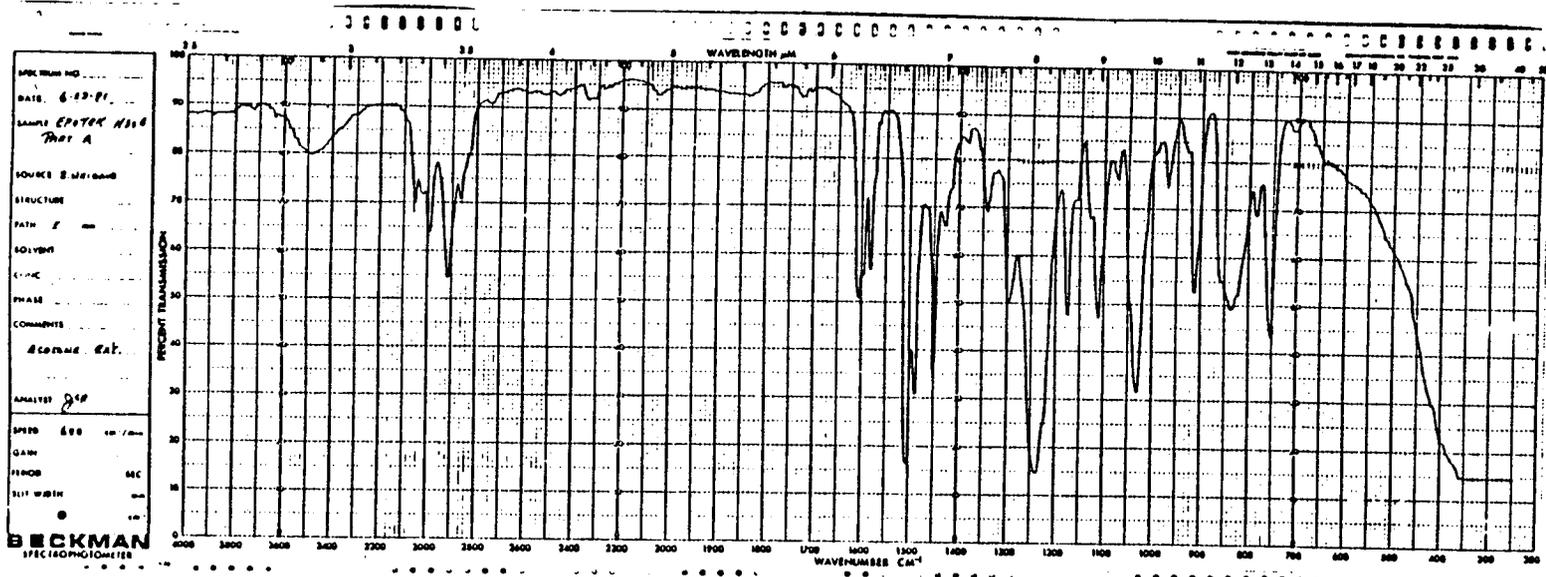


Figure 10. Infrared Spectrum of Epo-Tek H20E, Part A

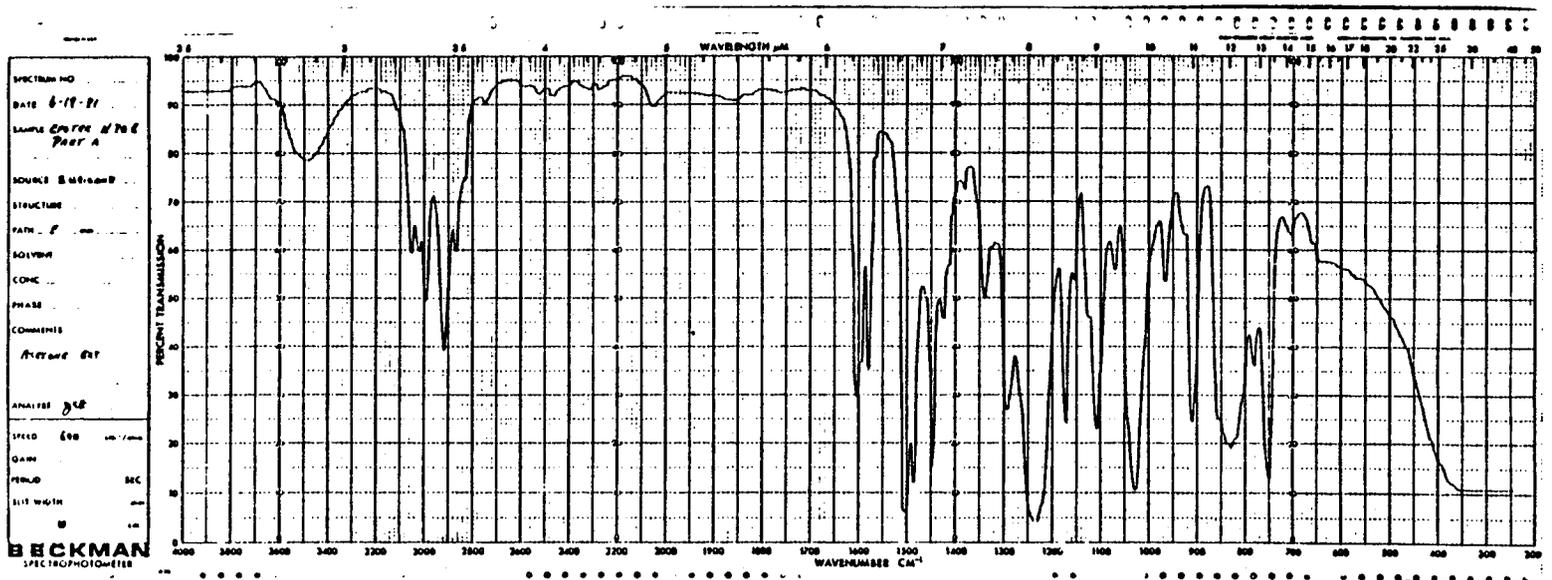


Figure 11. Infrared Spectrum of Epo-Tek H70E, Part A

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Figure 12. Infrared Spectrum of Epo-Tek H20E, Part B

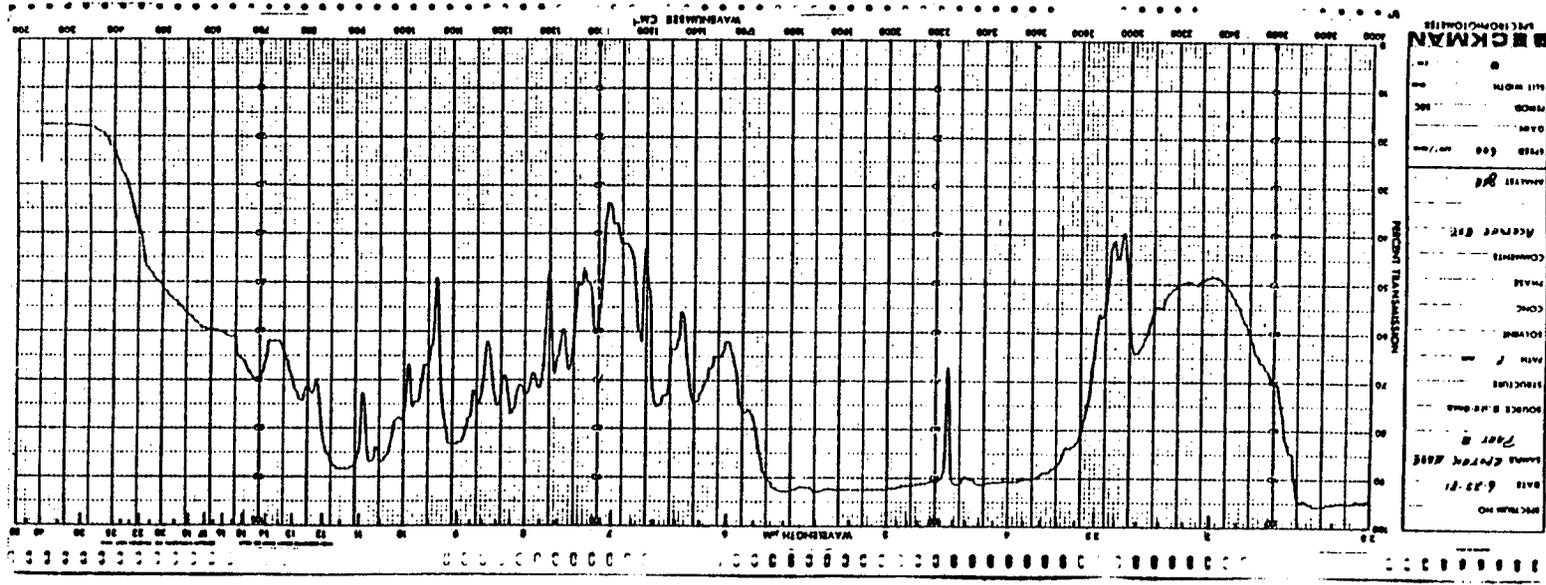
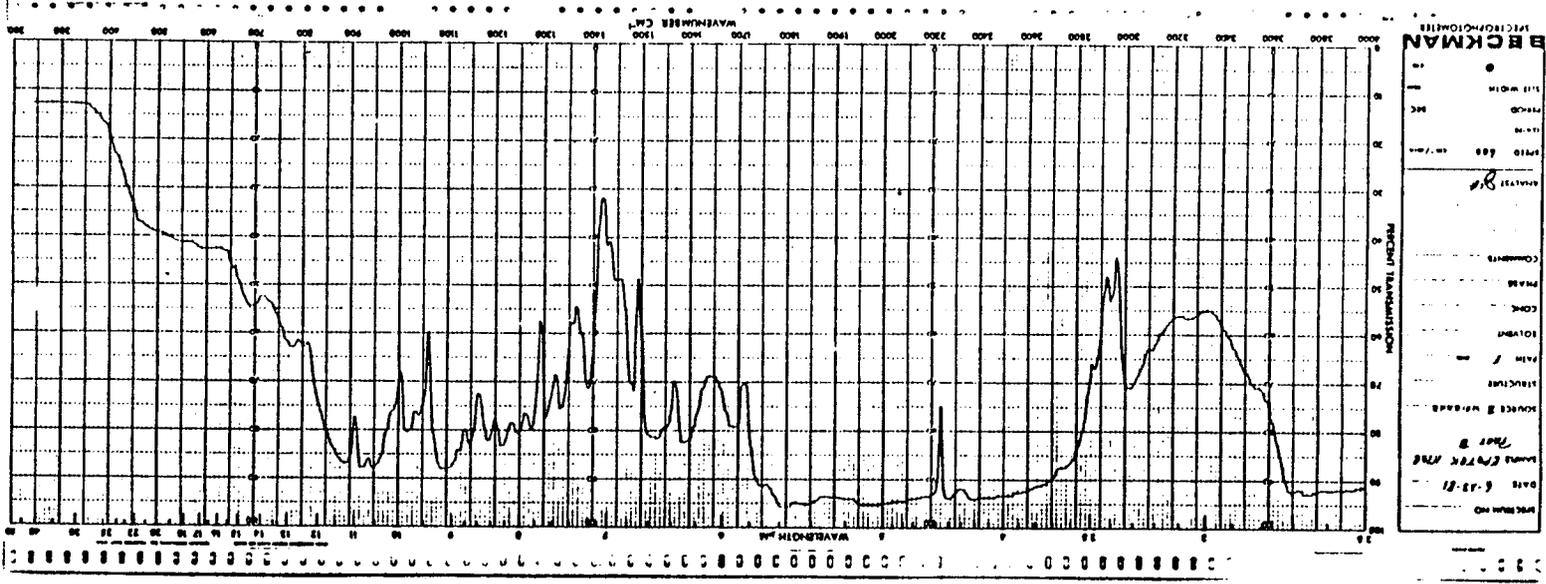


Figure 13. Infrared Spectrum of Epo-Tek H70E, Part B



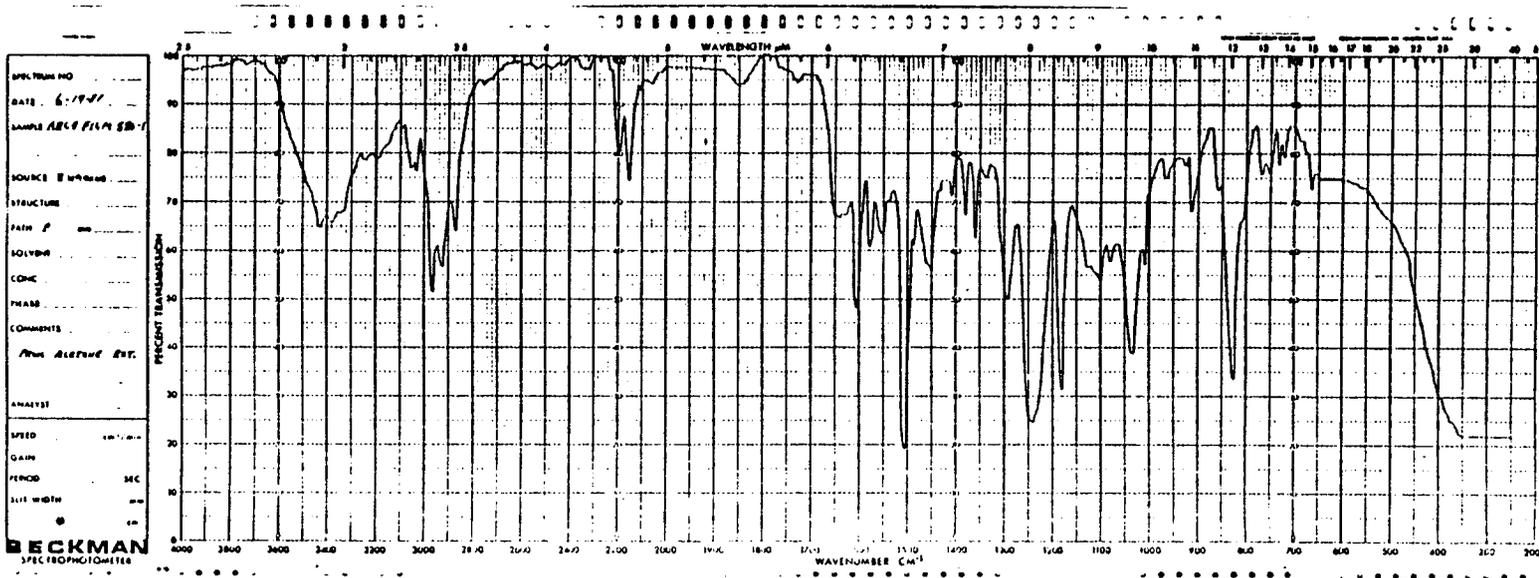


Figure 14. Infrared Spectrum of Ablefilm 550

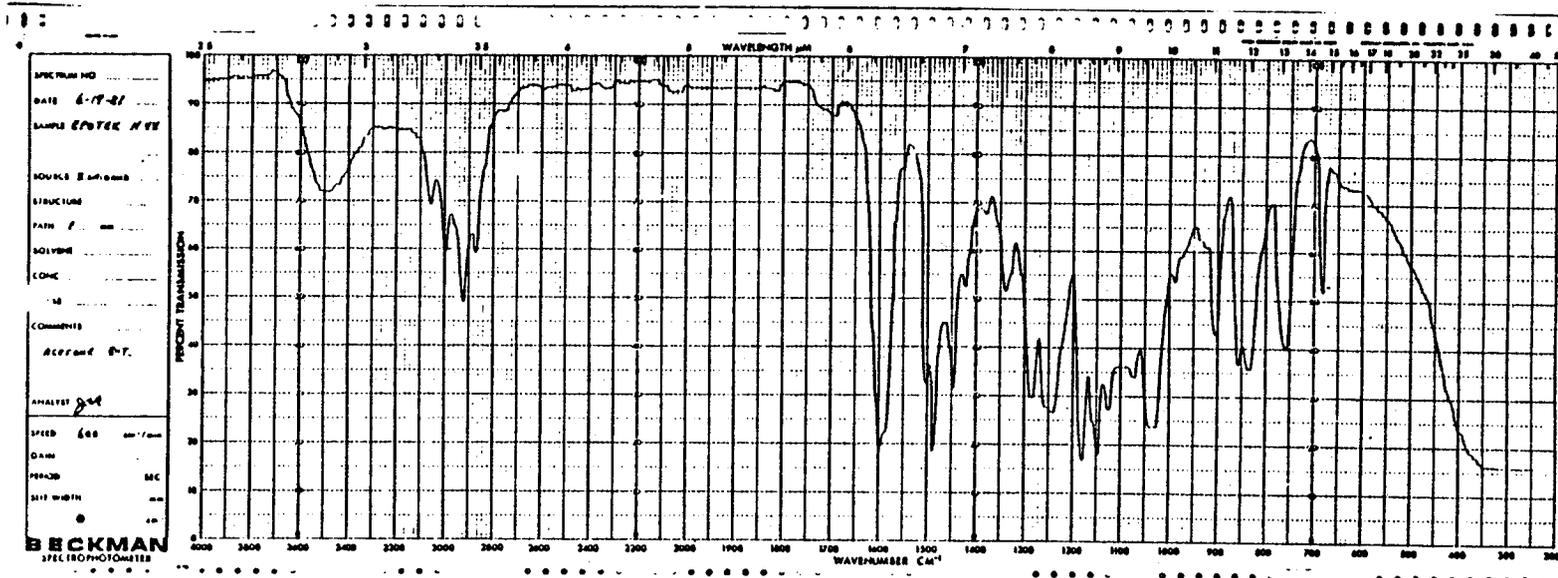
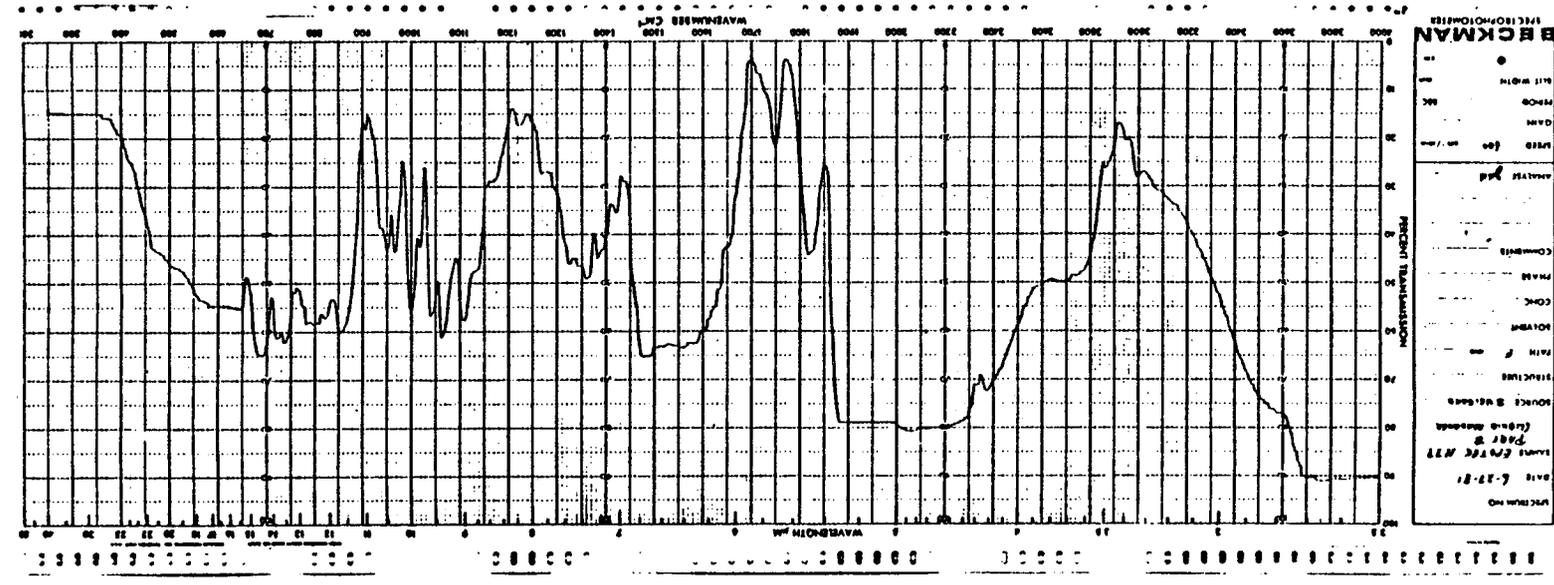
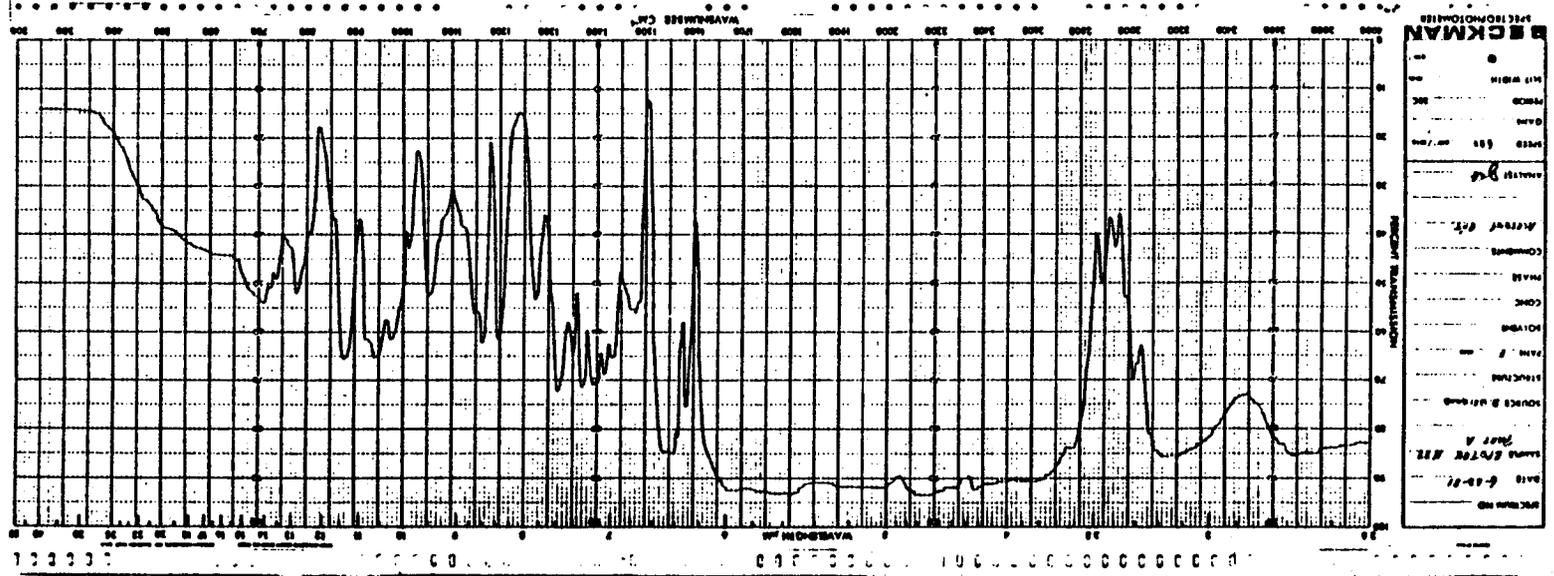


Figure 15. Infrared Spectrum of Epo-Tek H44

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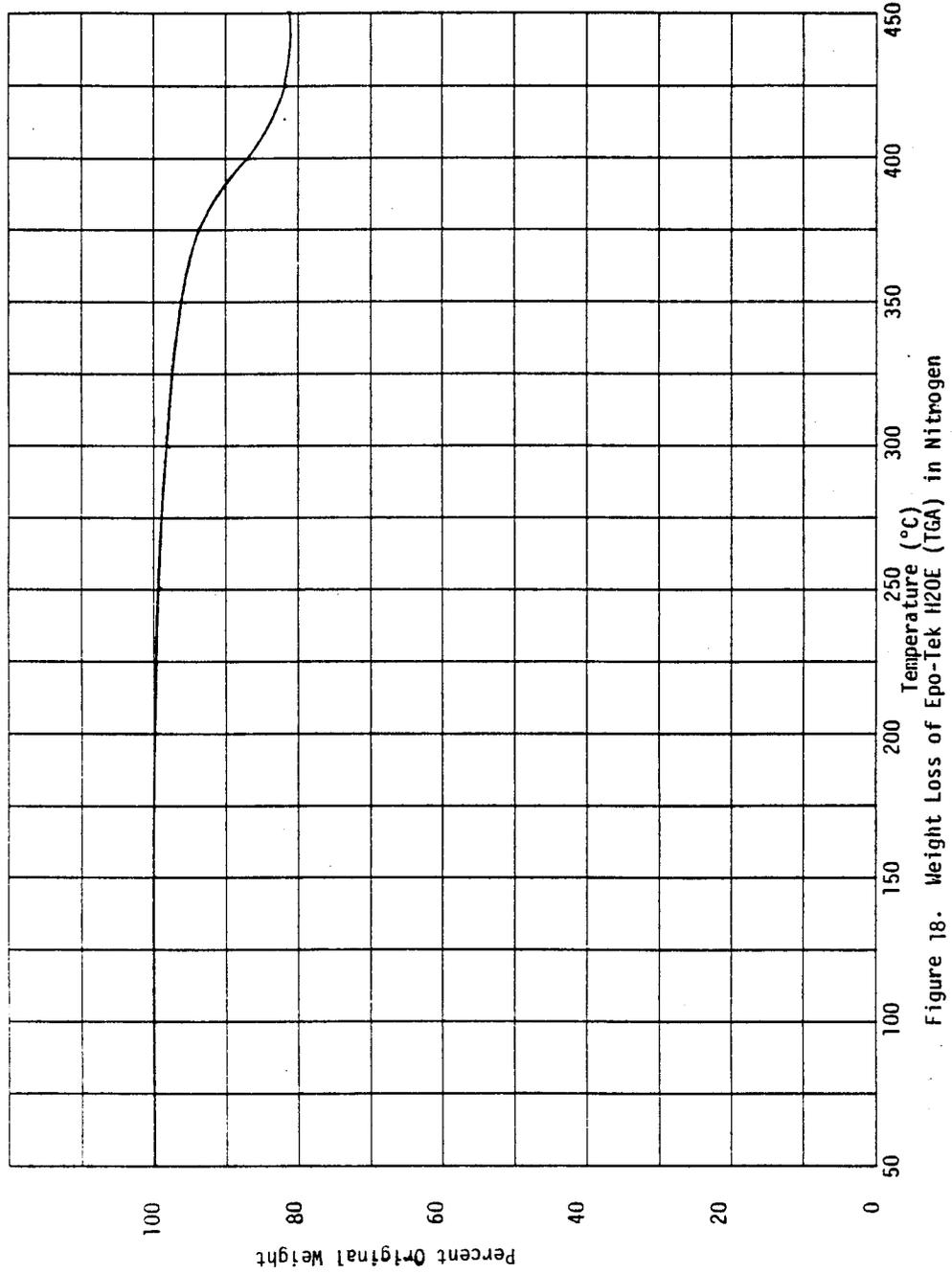


Figure 18. Weight Loss of Epo-Tek H20E (TGA) in Nitrogen

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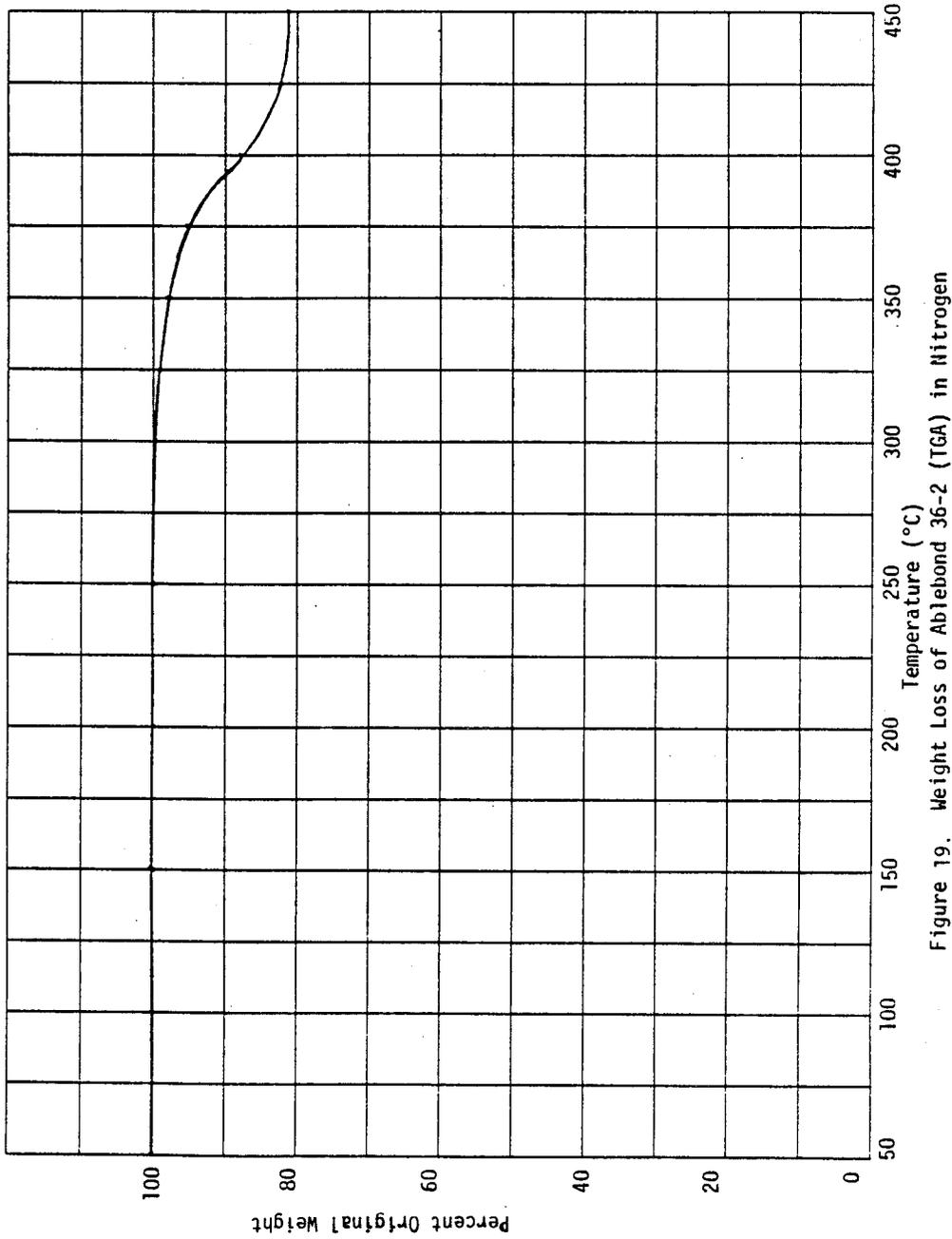


Figure 19. Weight Loss of Ablebond 36-2 (TGA) in Nitrogen

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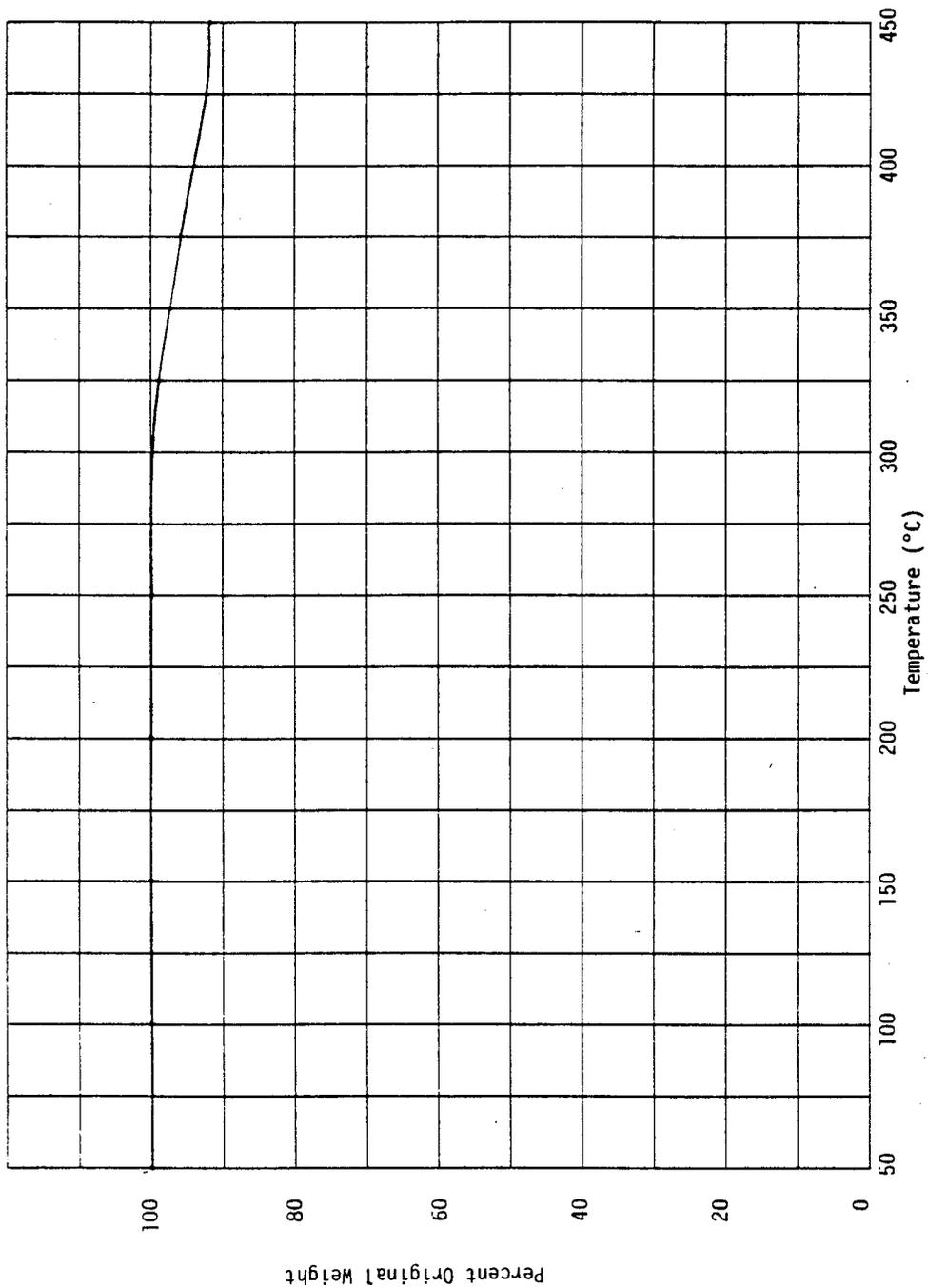


Figure 20. Weight Loss of Epo-Tek H44 (TGA) in Nitrogen

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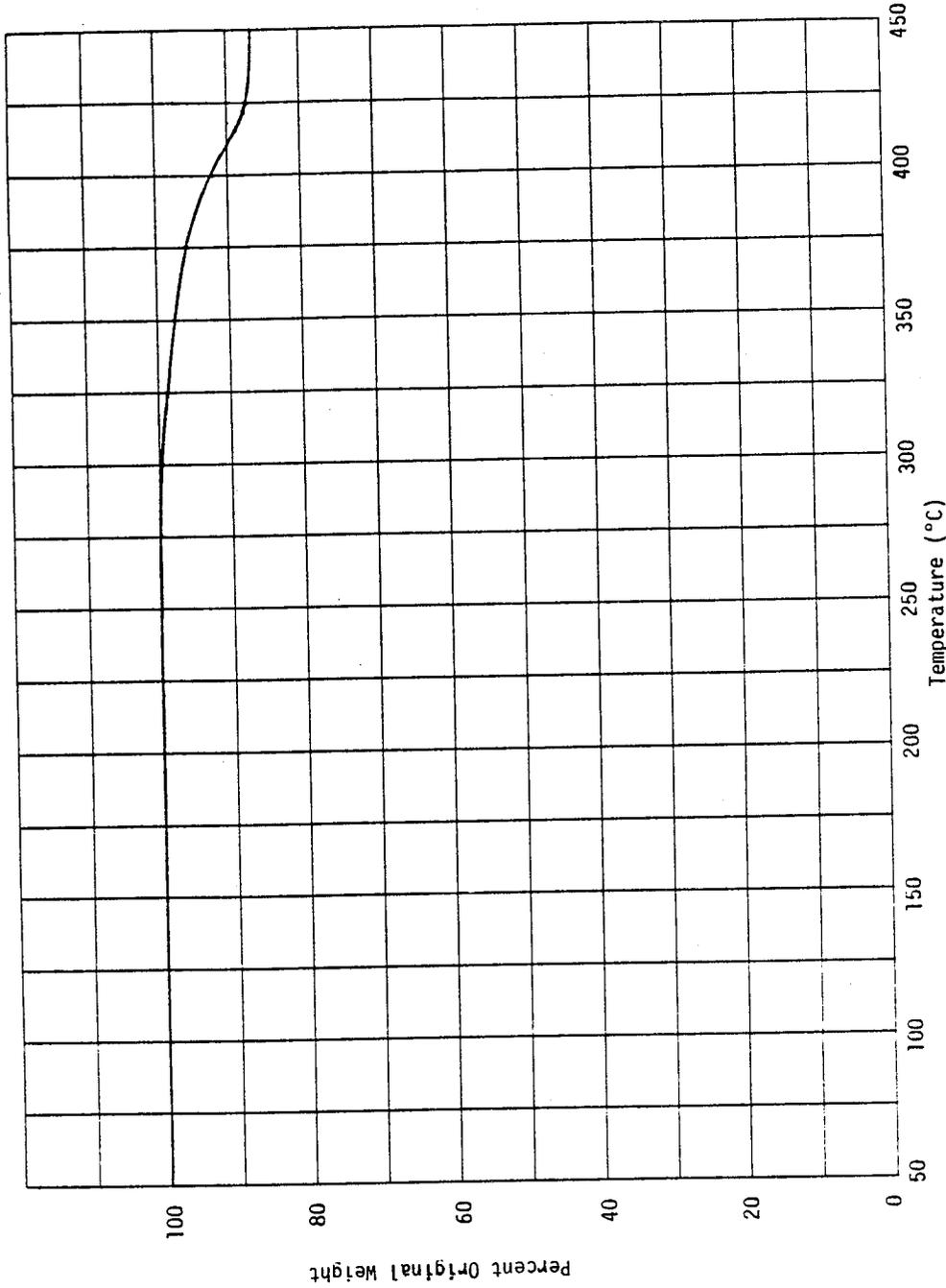


Figure 21 . Weight Loss of Ablebond 58-6 (TGA) in Nitrogen

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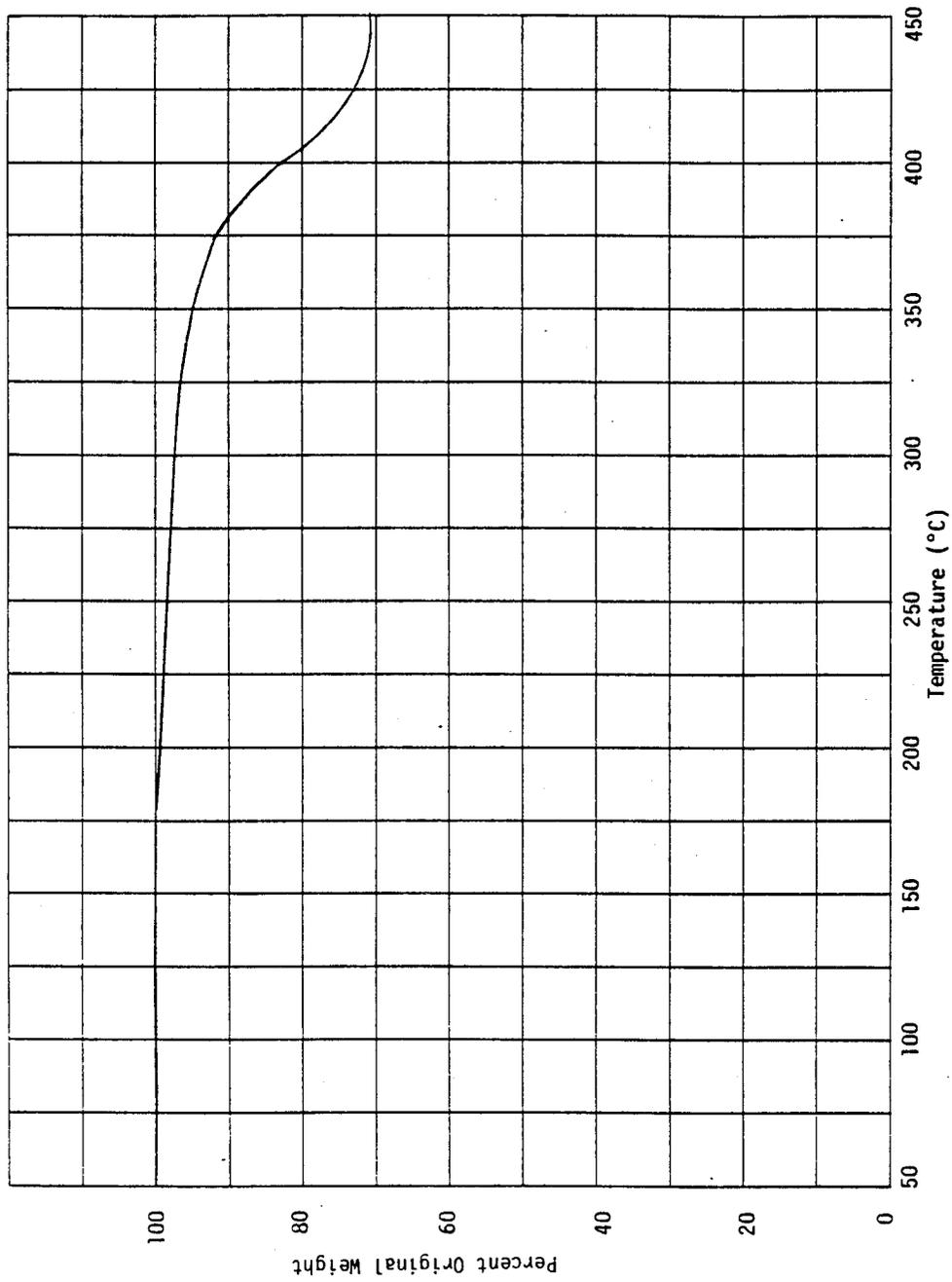


Figure 22. Weight Loss of Epo-Tek H70E (TGA) in Nitrogen
Cured 30 Minutes at 150°C

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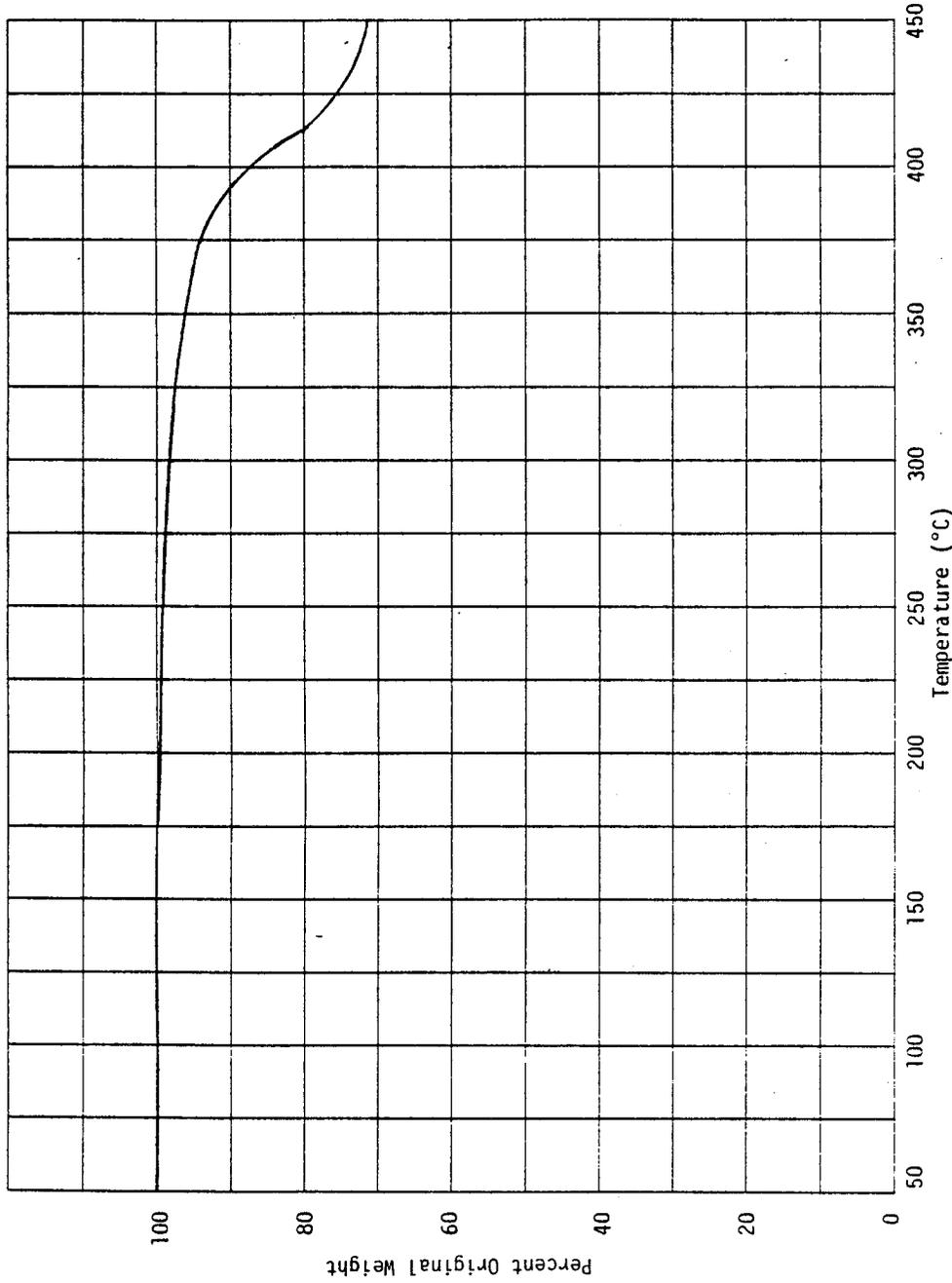


Figure 23. Weight Loss of Epo-Tek H70E (TGA) in Nitrogen Cured 60 Minutes at 150°C

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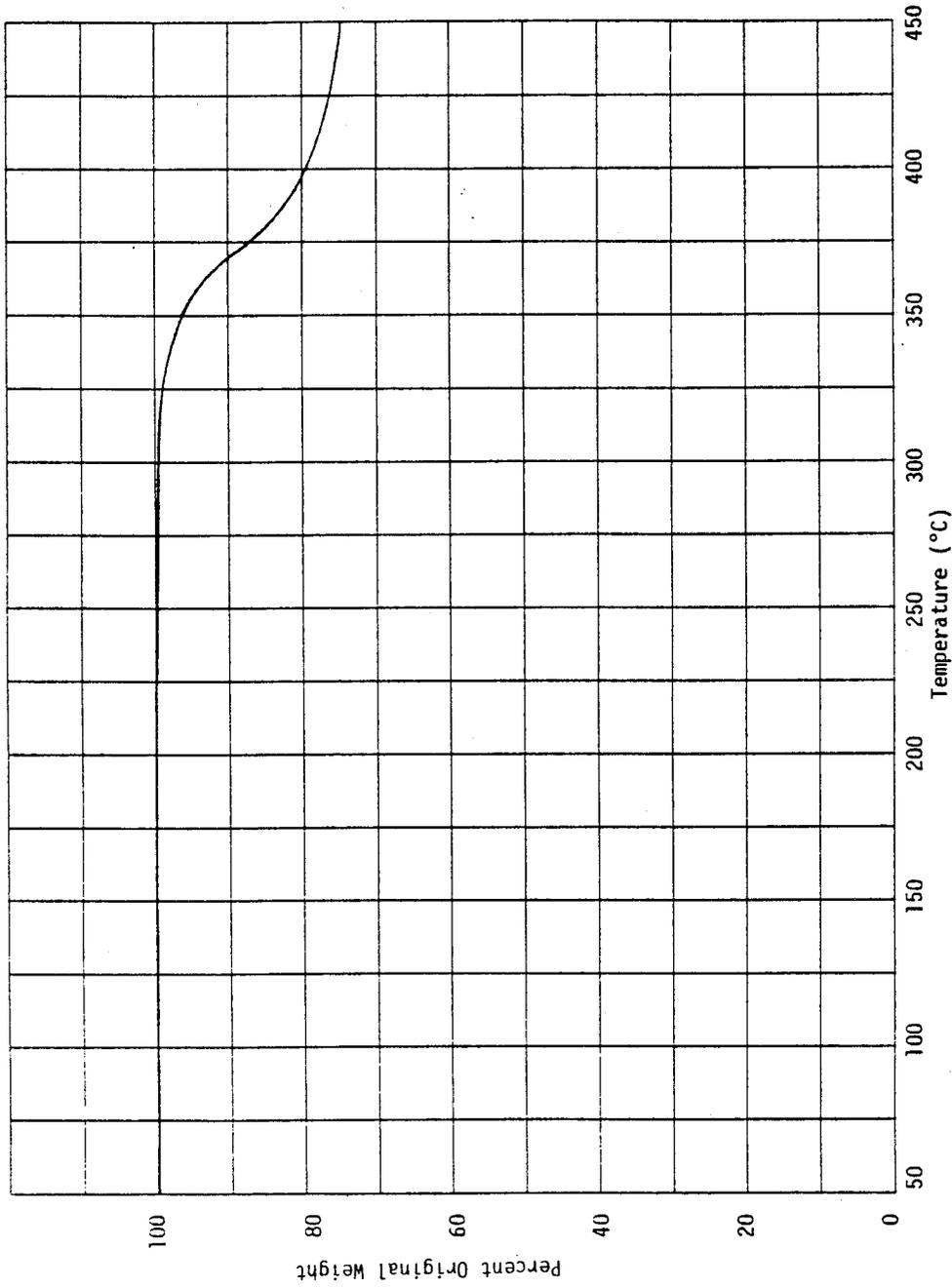


Figure 24 . Weight Loss of Epo-Tek H77 (TGA) in Nitrogen

Rockwell International

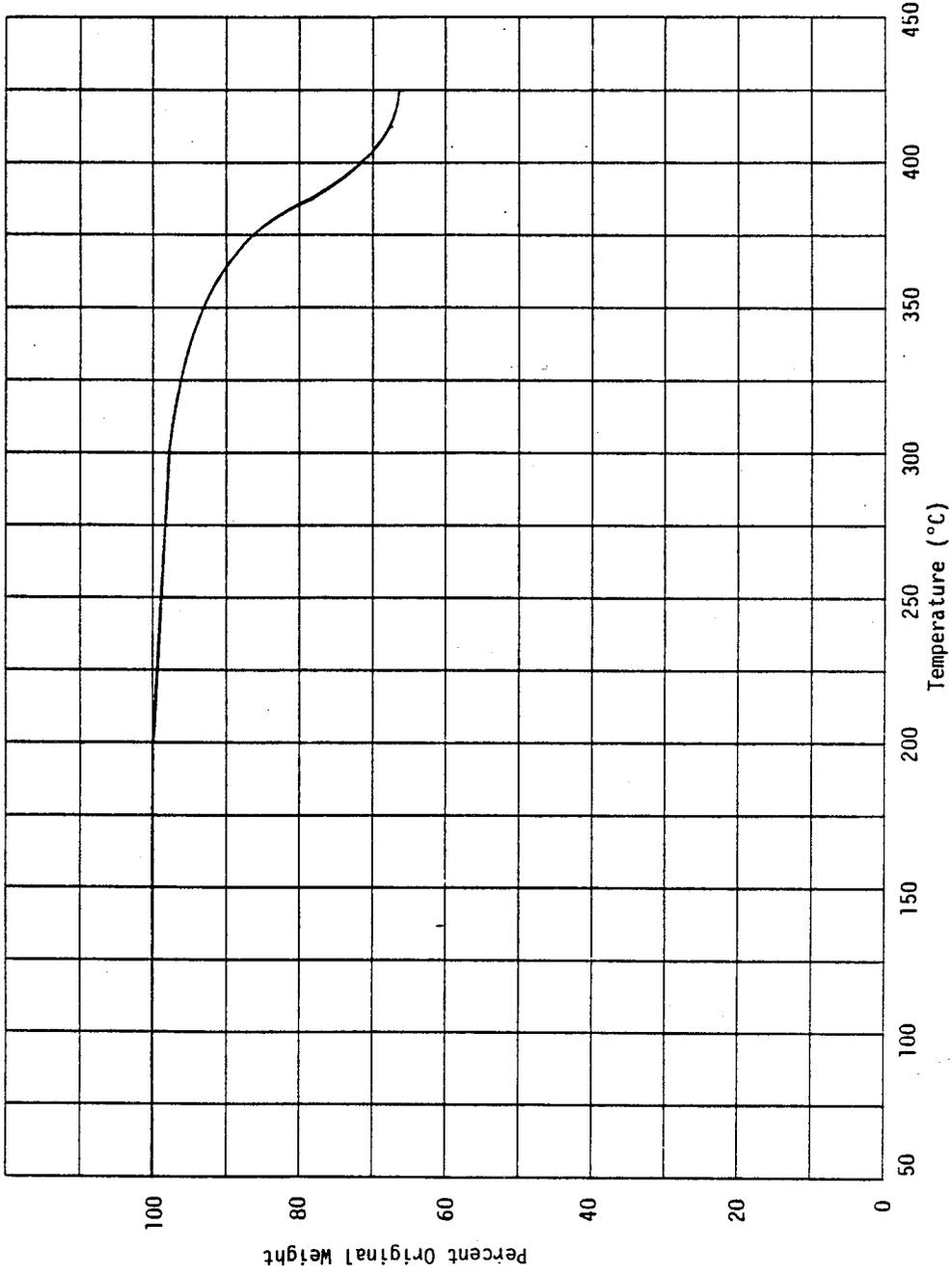


Figure 25 . Weight Loss of Ablebond 41-1 (TGA) in Nitrogen

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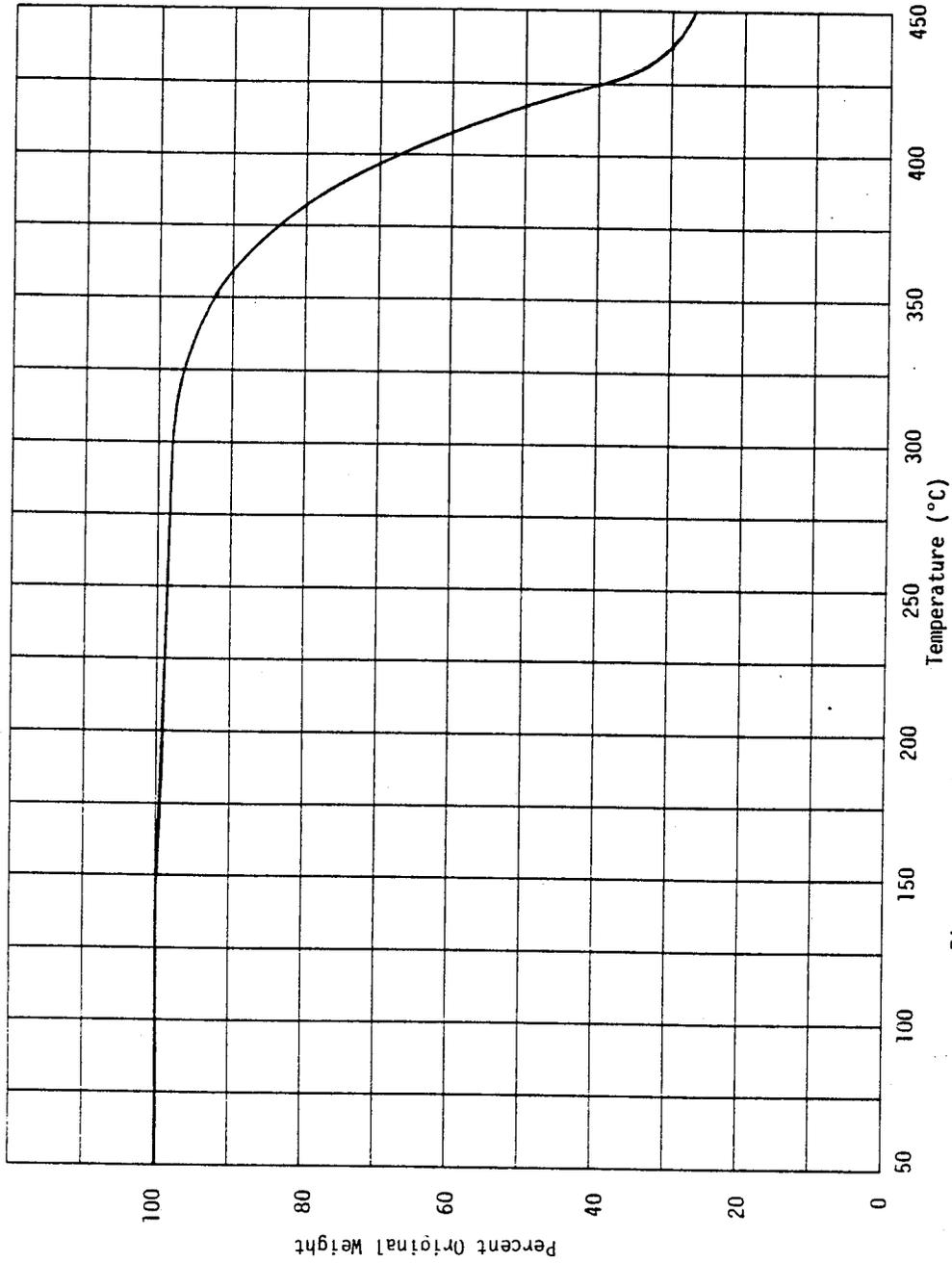


Figure 26. Weight Loss of Ablefilm 550 (TGA) in Nitrogen

TABLE 9. RESULTS OF WEIGHT LOSS (TGA) TESTS

Adhesives	Manufacturers' Recommended Cure (Hours @ 150°C)	Cure Schedule Used (Hours @ 150°C)	Weight Loss @ 250°C (%)
Epo-Tek H20E	0.083	1.0	0.8
Ablebond 36-2	0.5	1.0	0.0
Epo-Tek H44	0.5	1.0	0.0
Ablebond 58-6	0.5	1.0	0.1
Epo-Tek H70E	0.083	0.5 and 1.0	1.5 (1.0) ^a
Epo-Tek H77	0.5	1.0	0.3
Ablebond 41-1	0.5	1.0	1.0
Ablefilm 550	2.0	4.0	1.5

^aAfter 1 hour at 150°C

The water vapor content of the gases emitted from the adhesives was also determined in accordance with MIL-STD-883B, Method 1018.2, Procedure 1. Four test packages (2.54x2.54x0.279 cm) were prepared for each adhesive. Two of the packages were gold plated Kovar and two were nickel plated Kovar in each case. Only nickel plated lids were available for sealing the packages. To avoid schedule delays the nickel plated lids were used on all of the packages. The packages were leadless to prevent leakage problems at the glass seal but this will not be made a specification requirement. An example of the internal package configuration used for the nonconductive (substrate) adhesives is shown in Figure 27. The substrate dimensions are 1.91x1.91x0.064 cm. An example of the internal package configuration used for the electrically conductive adhesive is shown in Figure 28. A total of 22 devices (0.127x0.127 cm), calculated to be sufficient to cover approximately 10 percent of the area of a substrate normally used in a package of this size, were bonded directly to the package base with the adhesive to be tested. (This is assumed to be the average device density in a package of this size.) The adhesives were all cured at 150°C for double the time specified by the vendors as shown in Table 9. Following cure, the packages were vacuum-baked for 16 hours at 150°C at ≤ 6.67 pascals (believed to be the minimum requirement), hermetically sealed by seam welding in dry nitrogen and leak tested in accordance with MIL-STD-883B, Method 1014.1, Test Conditions A₁ and C₁. Prior to gas ambient analysis, the packages were baked at 125°C for 168 hours (to simulate burn-in) and allowed to stand for 24 hours at 25°C in air.

The results of the gas ambient analyses are shown in Tables 10 through 17. All four of the electrically conductive adhesives, Epo-Tek H20E and H44 and Ablebond 36-2 and 58-6, Tables 10 through 13 showed little or no water vapor in the packages. The amount of

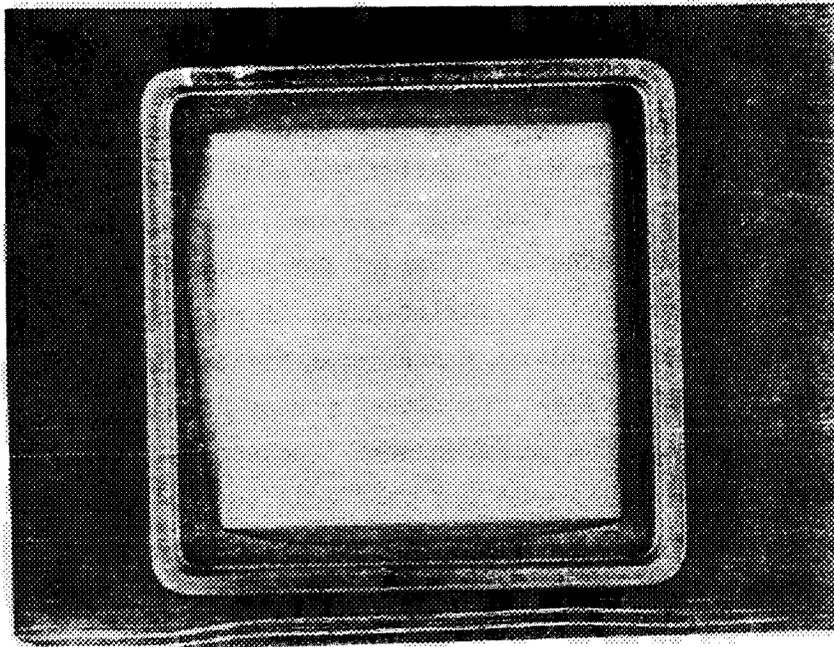


Figure 27. Package (Prior to Seal) Used for Gas Analysis of Nonconductive Adhesives

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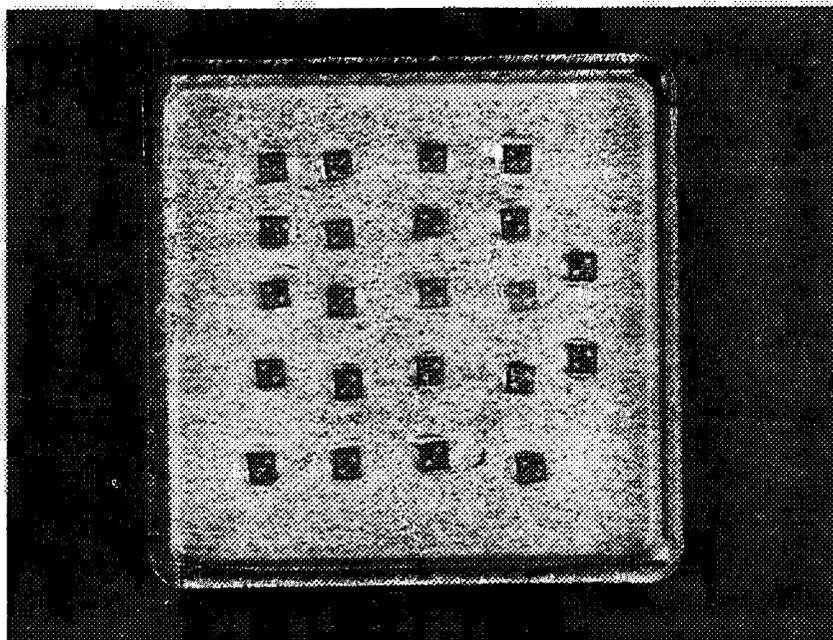


Figure 28. Package (Prior to Seal) Used for Gas Analysis of Electrically Conductive Adhesives

TABLE 10 • RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING EPO-TEK H2OE

AMU Specie	Concentration (% By Volume)			
	Gold Plated Packages	Nickel Plated Lids	Nickel Plated Packages	Nickel Plated Lids
	H2OEA1	H2OEA2	H2OEN1	H2OEN2
2 Hydrogen	0.003	ND ^a	ND ^a	ND ^a
4 Helium	ND ^a	ND ^a	ND ^a	ND ^a
15 Methane	0.011	0.008	0.012	0.009
18 Water Vapor	ND ^b	ND ^b	ND ^b	ND ^b
29 Nitrogen	99.939	99.957	99.968	99.966
32 Oxygen	ND ^a	ND ^a	ND ^a	ND ^a
40 Argon	0.013	0.008	0.001	0.010
44 Carbon Dioxide	0.034	0.027	0.019	0.015
17 Ammonia	ND ^b	ND ^b	ND ^b	ND ^b
31 Methanol	ND ^a	ND ^a	ND ^a	ND ^a
50 Methyl Chloride	ND ^a	ND ^a	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	ND ^a
Measured Leak Rate (scc/sec He)	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹
Volume of Gas @ 25°C (cc)	1.42	1.28	1.44	1.46

^aNone Detected, less than 0.001

^bNone Detected, less than 0.030

TABLE 11 . RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING EPO-TEK H44

AMU Specie	Concentration (% By Volume)			
	Gold Plated Packages	Nickel Plated Lids	Nickel Plated Packages	Nickel Plated Lids
	H44A1	H44A2	H44N1	H44N2
2 Hydrogen	0.001	ND ^a	ND ^a	ND ^a
4 Helium	ND ^a	ND ^a	ND ^a	ND ^a
15 Methane	0.012	0.011	0.013	0.014
18 Water Vapor	ND ^b	ND ^b	ND ^b	0.0750
29 Nitrogen	99.981	99.989	99.977	98.714
32 Oxygen	ND ^a	ND ^a	ND	1.084
40 Argon	ND ^a	ND ^a	0.002	0.058
44 Carbon Dioxide	0.006	ND ^a	0.008	0.055
17 Ammonia	ND ^b	ND ^b	ND ^b	ND ^b
31 Methanol	ND ^a	ND ^a	ND ^a	ND ^a
50 Methyl Chloride	ND ^a	ND ^a	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	ND ^a
Measured Leak Rate (scc/sec He)	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	7.6x10 ⁻⁷
Volume of Gas @ 25°C (cc)	1.45	1.37	1.50	1.34

^aNone Detected, less than 0.001
^bNone Detected, less than 0.030

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TABLE 12 . RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING ABLEBOND 36-2

AMU Specie	Concentration (% By Volume)			
	Gold Plated Packages	with Nickel Plated Lids	Nickel Plated Packages	Nickel Plated Lids
	36-2A1	36-2A2	36-2N1	36-2N2
2 Hydrogen	0.001	ND ^a	0.003	ND ^a
4 Helium	ND ^a	0.331	ND ^a	ND ^a
15 Methane	0.011	0.010	0.011	0.011
18 Water Vapor	ND ^b	0.2850	ND ^b	ND ^b
29 Nitrogen	99.964	97.814	99.971	99.970
32 Oxygen	ND ^a	1.403	ND ^a	ND ^a
40 Argon	ND ^a	0.066	0.001	0.001
44 Carbon Dioxide	0.024	0.091	0.014	0.018
17 Ammonia	ND ^b	ND ^b	ND ^b	ND ^b
31 Methanol	ND ^a	ND ^a	ND ^a	ND ^a
50 Methyl Chloride	ND ^a	ND ^a	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	ND ^a
Measured Leak Rate (scc/sec He)	<1.0x10 ⁻⁹	3.2x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹
Volume of Gas @ 25°C (cc)	1.18	1.55	1.38	1.54

^aNone Detected, less than 0.001

^bNone Detected, less than 0.030

TABLE 13. RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING ABLEBOND 58-6

AMU Specie	Concentration (% By Volume)			
	Gold Plated Packages	Nickel Plated Lids	Nickel Plated Packages	Nickel Plated Lids
	58-6A1	58-6A2	58-6N1	58-6N2
2 Hydrogen	0.001	0.004	ND ^a	ND ^a
4 Helium	ND ^a	ND ^a	ND ^a	ND ^a
15 Methane	0.012	0.010	0.011	0.010
18 Water Vapor	ND ^b	ND ^b	ND ^b	ND ^b
29 Nitrogen	99.927	99.919	99.944	99.938
32 Oxygen	ND ^a	ND ^a	ND ^a	ND ^a
40 Argon	0.001	0.001	ND ^a	0.001
44 Carbon Dioxide	0.059	0.066	0.045	0.051
17 Ammonia	ND ^b	ND ^b	ND ^b	ND ^b
31 Methanol	ND ^a	ND ^a	ND ^a	ND ^a
50 Methyl Chloride	ND ^a	ND ^a	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	ND ^a
Measured Leak Rate (scc/sec He)	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹
Volume of Gas @ 25°C (cc)	1.41	1.44	1.46	1.49

^aNone Detected, less than 0.001

^bNone Detected, less than 0.030

TABLE 14 . RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING EPO-TEK H77

AMU Specie	Concentration (% By Volume)			
	Gold Plated with Packages	Nickel Plated Lids	Nickel Plated Packages	Nickel Plated Lids
	H77A1	H77A2	H77N1	H77N2
2 Hydrogen	0.003	0.002	0.001	0.001
4 Helium	ND ^a	ND ^a	ND ^a	ND ^a
15 Methane	0.044	0.037	0.020	0.018
18 Water Vapor	ND ^b	ND ^b	ND ^b	ND ^b
29 Nitrogen	99.088	99.317	99.541	99.525
32 Oxygen	ND ^a	ND ^a	ND ^a	ND ^a
40 Argon	0.001	ND ^a	0.014	0.014
44 Carbon Dioxide	0.849	0.637	0.424	0.442
17 Ammonia	ND ^b	ND ^b	ND ^b	ND ^b
31 Methanol	ND ^a	ND ^a	ND ^a	ND ^a
50 Methyl Chloride	0.015	0.007	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	ND ^a
Measured Leak Rate (scc/sec He)	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹
Volume of Gas @ 25°C (cc)	1.18	1.19	1.31	1.34

^aNone Detected, less than 0.001

^bNone Detected, less than 0.030

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TABLE 15. RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING ABLEBOND 41-1

AMU Specie	Concentration (% By Volume)			
	Gold Plated Packages	Nickel Plated Lids	Nickel Plated Packages	Nickel Plated Lids
	41-1A1	41-1A2	41-1N1	41-1N2
2 Hydrogen	0.010	0.006	0.007	0.007
4 Helium	ND ^a	ND ^a	ND ^a	ND ^a
15 Methane	0.016	0.015	0.014	0.023
18 Water Vapor	2.5070	2.2830	2.3380	1.8900
29 Nitrogen	95.351	95.538	96.135	96.491
32 Oxygen	ND ^a	ND ^a	ND ^a	ND ^a
40 Argon	0.002	ND ^a	0.002	0.001
44 Carbon Dioxide	2.114	2.158	1.504	1.588
17 Ammonia	ND ^b	ND ^b	ND ^b	ND ^b
31 Methanol	ND ^a	ND ^a	ND ^a	ND ^a
50 Methyl Chloride	ND ^a	ND ^a	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	ND ^a
Measured Leak Rate (scc/sec He)	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹
Volume of Gas @ 25°C (cc)	1.28	1.13	1.36	1.25

^aNone Detected, less than 0.001
^bNone Detected, less than 0.030

TABLE 16. RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING ABLEFILM 550

AMU Specie	Concentration (% By Volume)			
	Gold Plated Packages	Nickel Plated Lids	Nickel Plated Packages	Nickel Plated Lids
	550A1	550A2	550N1	550N2
2 Hydrogen	0.021	0.013	0.021	0.015
4 Helium	ND ^a	ND ^a	ND ^a	ND ^a
15 Methane	0.073	0.060	0.078	0.072
18 Water Vapor	0.3070	0.2790	0.3100	0.2570
29 Nitrogen	98.489	99.001	98.750	98.828
32 Oxygen	ND ^a	ND ^a	ND ^a	ND ^a
40 Argon	0.001	ND ^a	ND ^a	0.001
44 Carbon Dioxide	0.182	0.122	0.110	0.137
17 Ammonia	0.805	0.420	0.629	0.549
31 Methanol	0.122	0.105	0.102	0.141
50 Methyl Chloride	ND ^a	ND ^a	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	ND ^a
Measured Leak Rate (scc/sec He)	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹
Volume of Gas @ 25°C (cc)	1.17	1.14	1.33	1.23

^aNone Detected, less than 0.001

TABLE 17. RESULTS OF GAS AMBIENT ANALYSES OF PACKAGES CONTAINING EPO-TEK H70E

AMU Specie	Concentration (% By Volume)			
	Gold Plated Packages	with Nickel Plated Lids	Nickel Plated Packages	with Nickel Plated Lids
	H70EA1	H70EA2	H70EN1	H70EN2
2 Hydrogen	0.005	0.007	0.016	0.037
4 Helium	0.767	ND ^a	ND ^a	ND ^a
15 Methane	0.018	0.012	0.057	0.107
18 Water Vapor	0.4570	0.0970	0.5180	0.7280
29 Nitrogen	97.644	99.619	98.484	97.470
32 Oxygen	0.691	ND ^a	ND ^a	ND ^a
40 Argon	0.073	0.010	0.012	0.041
44 Carbon Dioxide	0.223	0.071	0.181	0.346
17 Ammonia	0.122	0.184	0.661	1.082
31 Methanol	ND ^a	ND ^a	0.071	0.163
50 Methyl Chloride	ND ^a	ND ^a	ND ^a	ND ^a
91 Toluene	ND ^a	ND ^a	ND ^a	0.026
Measured Leak Rate (scc/sec He)	1.6x10 ⁻⁸	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹	<1.0x10 ⁻⁹
Volume of Gas @ 25°C (cc)	1.11	1.26	1.33	1.28

^aNone Detected, less than 0.001

carbon dioxide in the packages was also quite low. The only packages showing detectable water vapor (≥ 300 ppm) were H44N2 (Table 11), and 36-2A2 (Table 12) which were shown to be leakers by the presence of oxygen/helium.

In the case of the nonconductive adhesives only Epo-Tek H77 (Table 14) showed no detectable water vapor. The carbon dioxide content of the H77 packages was approximately an order of magnitude higher than that of the packages containing electrically conductive adhesives, however, and the gold-plated packages with nickel-plated lids showed traces of methyl chloride (70 and 150 ppm) while the entirely nickel-plated packages showed less than 10 ppm, the limit of detectability. The packages containing Ablebond 41-1 (Table 15) showed the highest water vapor content (18,900 ppm to 25,070 ppm) and the highest carbon dioxide content (15,040 ppm to 21,580 ppm). A much longer vacuum bake would be required to reduce the water vapor content to the MIL-STD-883 requirement of ≤ 5000 ppm. The packages containing Ablefilm 550 (Table 16) showed an acceptable water vapor content of 2570 to 3100 ppm but showed high levels of ammonia (4,200 to 8,050 ppm) and methanol (1,020 to 1,410 ppm). The ammonia, which is basic, can cause corrosion of aluminum metalization while the methanol can serve both as a freezing point depressant for water and as a conductive medium for the ammonia or ionic impurities increasing the corrosion potential. A much longer vacuum bake and/or higher cure temperature and time would again be required for the Ablefilm 550 to reduce the ammonia and methanol levels. The packages containing Epo-Tek H70E (Table 17) show a considerable variation in the contents of the gas ambients. While package H70EA1 shows an acceptable leak rate of 1.6×10^{-8} scc/sec He, it is an obvious leaker based on the helium, oxygen and argon contents. Comparison of the results for package H70EA2 which is gold-plated with a nickel-plated lid with those for packages H70EN1 and H70EN2 which are entirely nickel-plated

shows a higher water vapor, carbon dioxide and ammonia content for the nickel-plated packages and the presence of methanol only in the nickel-plated packages. Package H70EN2 also showed 260 ppm of toluene. A larger sample size would be required to determine if there is a significant difference in the results for the gold and nickel-plated packages. The results do indicate, however, that the sealing process requires further development to reduce the water vapor, ammonia, and methanol contents.

Infrared analysis, Figures 6 and 7, showed Epo-Tek H20E and H70E to be identical except for the fillers used. The H20E, Table 10, showed no detectable water vapor or ammonia while the H70E, Table 17, showed significant amounts of both materials. The infrared spectra of Ablebond 36-2, 58-6 and 41-1, Figures 3 through 5, also showed them to be identical except for the fillers. The Ablebond 36-2 and 58-6 (Tables 12 and 13) showed no detectable water vapor and minor amounts of carbon dioxide while Ablebond 41-1 (Table 15) showed significant amounts of both. It is therefore apparent that the filler can significantly affect the outgassing of the adhesive.

A comparison of weight loss measurements based on thermogravimetric analysis (TGA) and water vapor emission based on mass spectrometric analysis (MS) are shown in Table 18. It can be seen that a good correlation exists for the acceptable adhesives (TGA: \leq 0.3 wt%, MS \leq 5000 ppm) with the exception of Epo-Tek H20E which would fail the TGA requirement. Longer cures and/or higher cure temperatures would undoubtedly allow all of the adhesives to meet the TGA requirement. Similarly, longer cures and/or higher cure temperatures combined with a longer vacuum bake and/or higher bake temperature, would probably allow all of the adhesives to meet the water vapor requirement. (The cure temperature/vacuum bake temperature are limited to \leq 165°C as now proposed in the NASA specification.) Since the objective of this specification is

TABLE 18. COMPARISON OF WEIGHT LOSS MEASUREMENT PROCEDURES

<u>Adhesive</u>	<u>Weight Loss at 250°C (TGA)</u> <u>(grams/gram x 100)</u>	<u>Water Vapor (MS)</u> <u>ppm</u>
Epo-Tek H20E	0.8	<300
Ablebond 36-2	0.0	<300
Epo-Tek H44	0.0	<300
Ablebond 58-6	0.0	<300
Epo-Tek H70E	1.5 ^a (1.0) ^b	4,477 ^{c,d}
Epo-Tek H77	0.3	<300
Ablebond 41-1	1.0	22,545
Ablefilm 550	1.3	2,883 ^{c,d}

^aCured 0.5 hour at 150°C

^bCured 1.0 hour at 150°C

^cMethanol also present

^dAmmonia also present

to establish such parametric data, it is recommended that both tests be retained in the specification and that MS limits be established for ammonia, methanol, and other outgassed materials which may be detrimental. Until calibration procedures are established, however, these limits will have to be set as reference values only. The test facility will be required to record and report interpreted scan data over the mass range of 2 to 100. This will allow the procuring activity and manufacturer to establish acceptance levels.

3.4.4 BOND SHEAR STRENGTH TESTS

Bond shear tests were conducted using semiconductor die, chip capacitors, and ceramic chips (to represent substrate attachment) as shown in Table 19. Five devices of each type were shear tested under five different conditions as follows:

1. At 25°C
2. At 150°C
3. At 25°C after solvent immersion
4. At 25°C after 200 cycles from -65°C to 150°C
5. At 25°C after 1000 hours at 150°C

The solvent immersion test consisted of consecutively immersing the test specimens for one-half hour each in isopropyl alcohol, a solution of 50 vol percent methylene chloride in trichlorotrifluoroethane and in 100 percent trichlorotrifluoroethane. The specimens were allowed to dry for a minimum of three hours prior to shear testing. Temperature cycling was performed in accordance with MIL-STD-883B, Method 1010.4, Test Condition C.

Test specimens representative of those used for semiconductor (transistor) die testing are shown in Figures 29 and 30. Five die each, bonded with two adhesives, are contained on each substrate. Test specimens used for capacitor shear testing are shown in Figures 31 and 32, and a substrate shear test specimen is shown in Figure 33. Package lids were used as substitutes for the package base in the latter case.

TABLE 19. DEVICES AND SUBSTRATES SELECTED FOR BOND SHEAR TESTS

Device Type	Contact Metallization	Size (cm)	Weight (Kg) ^a	Substrate	Metallization
Transistor	Alloyed Gold	0.051 x 0.051	1.0×10^{-7}	99.5% Alumina	Gold Thin Film
Transistor	Alloyed Gold	0.102 x 0.102	6.5×10^{-7}	99.5% Alumina	Gold Thin Film
Capacitor	Silver	0.127 x 0.102 x 0.102	4.2×10^{-6}	99.5% Alumina	Gold Thin Film
Capacitor	Silver	0.699 x 0.216 x 0.152	1.34×10^{-4}	96% Alumina	Gold Thick Film
Substrate Chip	None	0.127 x 0.127 x 0.038	2.3×10^{-6}	Kovar	Plated Gold

^aAverage of ten devices in each case.

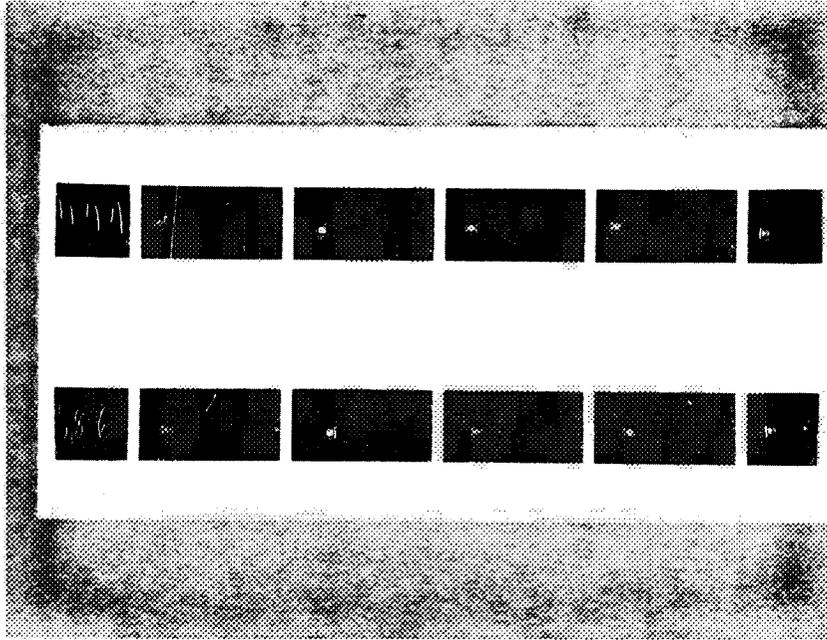


Figure 29. Small Semiconductor Die Shear Test Specimen

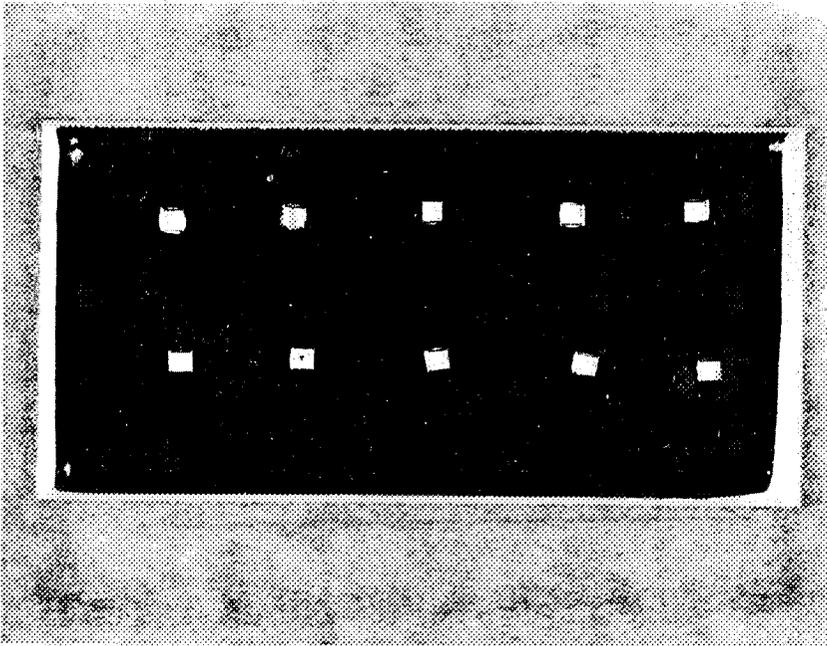


Figure 30. Large Semiconductor Die Shear Test Specimen

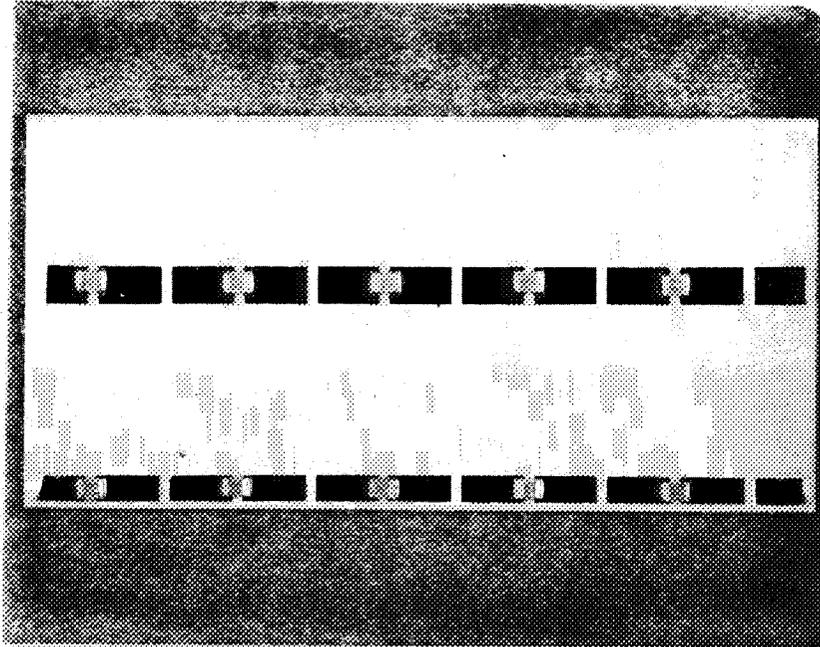


Figure 31. Small Capacitor Shear Test Specimen

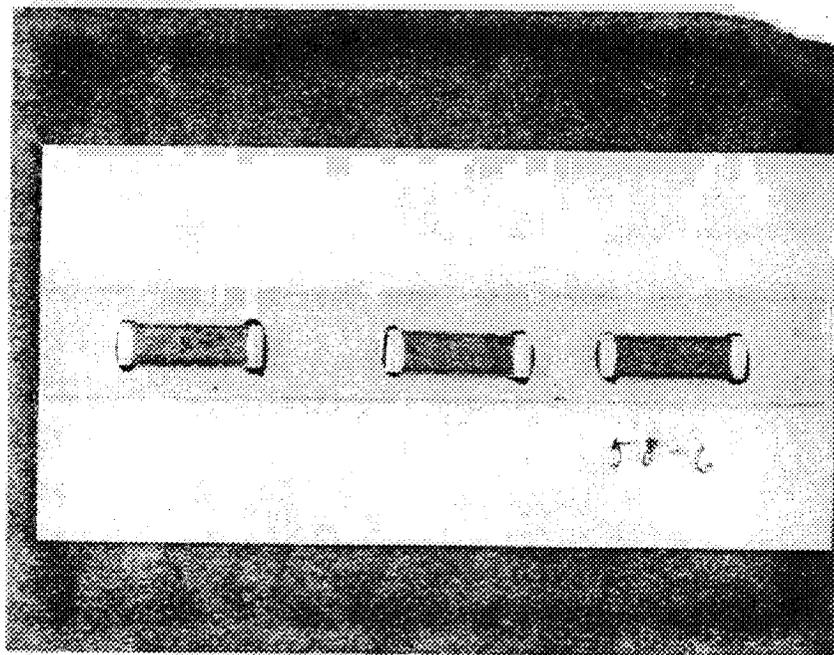


Figure 32. Large Capacitor Shear Test Specimen

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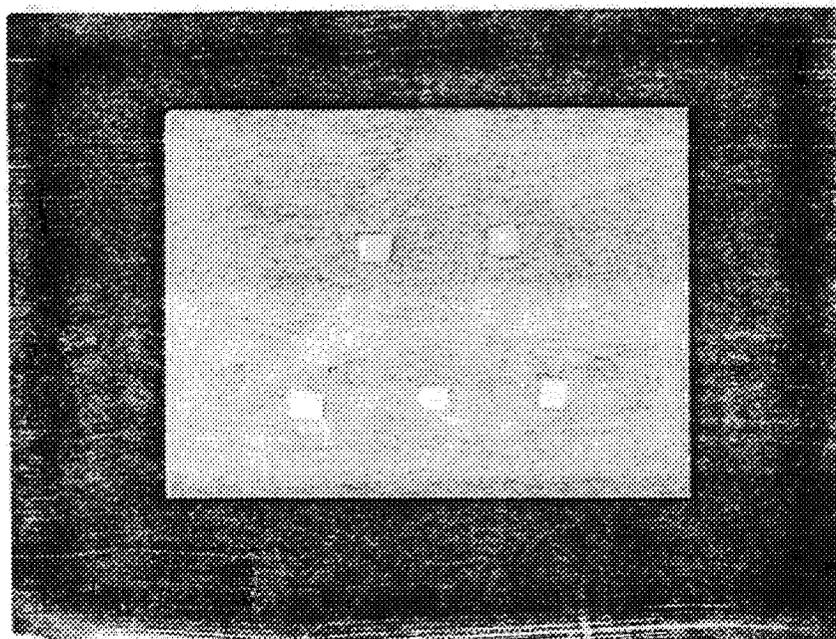


Figure 33. Substrate Shear Test Specimen

All substrates were cleaned prior to bonding by scrubbing consecutively in deionized water, isopropyl alcohol, and trichlorotrifluoroethane followed by rinsing with clean trichlorotrifluoroethane and blowing dry with dry nitrogen. All filleting was removed from around the semiconductor die and substrate chips prior to cure. The fillets were not removed from the capacitors, however, and only the minimal amount of adhesive was used (see Figure 32) to obtain a worst case shear condition.

The results of the shear tests are shown in Tables 20 through 24 and Figures 34 through 38. The NASA specification requires a bond strength $\geq 10S$ calculated using the following equation with a minimum requirement of 6.9×10^6 newtons/meter² (1000 psi):

$$S = \frac{9.8Wa}{A}$$

where: S = bond strength (newtons/meter²)

W = weight of device or substrate expressed
in kilograms

a = constant acceleration requirement per
MIL-STD-883, Method 2001 in Gs

A = bond area expressed in square meters

9.8 = gravitational constant expressed in
meters/second²

The calculated bond strength requirement for each of the devices used, assuming a constant acceleration requirement of 20,000 Gs, is included in the tables and in the figures. In the case of the transistors, all of the electrically conductive adhesives met the calculated requirements (Tables 20 and 21 and Figures 34 and 35) but Epo-Tek H20E showed an average shear strength of 5.3 meganewtons/meter² (750 psi) at 150°C for the 0.102 x 0.102 cm transistors failing the minimum requirement of 6.9 meganewtons/meter² (1000 psi).

In the case of the electrically insulative adhesives used

TABLE 20. COMPARISON OF BOND SHEAR STRENGTH RESULTS FOR SMALL TRANSISTORS (0.051 x 0.051 cm)

Adhesive	Shear Strength												Calculated Requirement ^a					
	At 25°C			At 150°C			At 25°C After Solvent Immersion			At 25°C After 200 Temp Cycles						At 25°C After 1000 hr At 150°C		
	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI
Ablebond 36-2	12	47	6900	6.7	26	3800	18	69	10000	14	54	7800	16	60	8800	0.20	0.75	110
	15	56	8100	8.9	34	5000	21	81	11700	20	77	11200	10	39	5600			
	13	52	7500	8.9	34	5000	20	77	11200	12	46	6700	29	111	16000			
	18	69	10000	5.6	22	3100	22	85	12300	4.5	17	2500	12	47	6900			
	12	47	6900	4.4	17	2500	8.9	34	4900	13	50	7300	16	60	8800			
Average	14	54	7900	6.9	27	3900	18	69	10000	13	50	7300	17	64	9300			
Epo-Tek H20E	12	47	6900	1.1	4	600	11	42	6100	5.6	22	3200	17	64	9400	0.20	0.75	110
	16	60	8800	2.2	9	1300	20	77	11200	11	42	6100	21	81	11900			
	13	52	7500	2.2	9	1300	14	54	7800	16	62	9000	17	64	9400			
	20	77	11300	2.2	9	1300	16	62	9000	17	65	9400	5.6	22	3100			
	16	60	8800	2.2	9	1300	8.9	34	4900	12	46	6700	36	137	20000			
Average	15	59	8600	2.0	8	1100	14	54	7800	12	46	6700	19	73	10800			
Ablebond 58-6	5.6	22	3100	5.6	22	3100	13	50	7300	5.6	22	3200	18	68	10000	0.20	0.75	110
	8.9	34	5000	3.3	13	1900	11	42	6100	8.9	34	4900	12	47	6900			
	7.8	30	4400	3.3	13	1900	7.8	30	4400	4.5	17	2500	8.9	34	5000			
	13	52	7500	4.4	17	2500	22	85	12300	7.8	30	4400	5.6	22	3100			
	11	43	6300	4.4	17	2500	20	77	11200	11	42	6100	11	47	6300			
Average	9.3	36	5300	4.2	16	2400	15	57	8300	7.6	29	4200	11	47	6300			
Epo-Tek H44	7.8	30	4400	3.3	13	1900	8.9	34	5000	3.3	13	1900	13	51	7500	0.20	0.75	110
	13	52	7500	4.4	17	2500	7.8	30	4400	17	65	9400	15	56	8100			
	8.9	34	5000	11	43	6300	8.9	34	5000	8.9	34	4900	12	47	6900			
	6.7	26	3800	4.4	17	2500	14	54	7800	5.6	22	3200	12	47	6900			
	11	43	6300	7.8	30	4400	11	42	6100	10	38	5500	12	47	6900			
Average	9.6	37	5400	6.2	24	3500	10	39	5700	8.9	34	4400	13	50	7300			

^aBased on an acceleration of 20,000 Gs

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TABLE 21. COMPARISON OF BOND SHEAR STRENGTH RESULTS FOR INTERMEDIATE SIZE TRANSISTORS (0.102 x 0.102 cm)

Adhesive	Shear Strength												Calculated Requirement ^a					
	At 25°C			At 150°C			At 25°C After Solvent Immersion			At 25°C After 200 Temp Cycles						At 25°C After 1000 hrs At 150°C		
	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI
Ablebond 36-2	27	26	3800	27	26	3800	31	30	4400	16	16	2300	53	51	7400	1.3	1.2	180
	22	21	3000	11	11	1600	27	26	3800	42	41	5900	18	17	2500			
	62	60	8700	17	17	2500	18	17	2500	41	40	5800	45	44	6400			
	62	60	8700	31	30	4400	27	26	3800	51	50	7300	36	35	5100			
	53	51	7400	22	21	3000	36	35	5100	28	27	3900	49	48	7000			
	Average	45	44	6400	22	21	3000	28	27	3900	36	35	5100	45	44			
Epo-Tek H20E	13	12	1700	5.6	6	780	22	21	3100	75	73	10600	46	45	6500	1.3	1.2	180
	22	21	3000	6.7	7	940	31	30	4400	27	26	3800	61	59	8600			
	36	35	5100	5.6	6	780	22	21	3100	13	12	1700	42	41	5900			
	45	42	6100	4.4	4	630	22	21	3000	34	33	4800	31	30	4400			
	36	35	5100	4.4	4	630	45	44	6400	67	65	9400	45	44	6400			
	Average	30	29	4200	5.3	5	750	28	27	3900	43	42	6100	44	43			
Ablebond 58-6	67	65	9400	8.9	9	1300	36	35	5100	31	30	4400	24	23	3300	1.3	1.2	180
	53	51	7400	19	18	2600	53	51	7400	17	16	2300	48	47	6800			
	45	44	6400	25	24	3500	31	30	4400	50	49	7100	72	70	10100			
	8.9	8.6	1200	11	11	1600	89	86	12000	45	44	6400	77	75	10900			
	22	21	3000	16	16	2300	53	51	7400	19	18	2600	71	69	10000			
	Average	39	38	5500	16	16	2300	52	50	7300	32	31	4500	58	56			
Epo-Tek H44	22	21	3000	45	44	6400	49	48	7000	34	33	4800	50	49	7100	1.3	1.2	180
	45	44	6400	18	17	2500	22	21	3000	29	28	4100	45	44	6400			
	22	21	3000	30	29	4200	18	17	2500	28	27	3900	30	29	4200			
	18	17	2500	23	22	3200	27	26	3800	19	18	2600	22	21	3000			
	18	17	2500	28	27	3900	22	21	3000	23	22	3200	45	44	6400			
	Average	25	24	3500	29	28	4100	28	27	3900	27	26	3800	38	37			

^aBased on an acceleration of 20,000 Gs

TABLE 22. COMPARISON OF BOND SHEAR STRENGTH RESULTS FOR SUBSTRATE CHIPS (0.127 x 0.127 x 0.038 cm)

Adhesive	Shear Strength																	
	At 25°C			At 150°C			At 25°C After Solvent Immersion			At 25°C After 200 Temp Cycles			At 25°C After 1000 hrs At 150°C			Calculated Requirement ^a		
	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI
Epo-Tek H70E	62	39	5700	8.9	5.6	800	49	31	4500	89	56	8100	156	98	14200	4.6	2.8	400
	49	31	4500	2.2	1.4	200	49	31	4500	71	44	6400	160	100	14500			
	45	28	4100	5.6	3.5	500	76	48	7000	89	56	8100	80	50	7300			
	62	39	5700	4.5	2.8	400	53	33	4800	102	64	9300	151	94	13600			
	58	36	5200	4.5	2.8	400	45	28	6500	80	50	7300	98	61	8800			
	55	34	4900	5.3	3.3	500	54	34	4900	86	54	7800	129	81	11700			
Epo-Tek H77	138	86	12500	11.1	6.9	1000	80	50	7300	134	84	12200	22	14	2000	4.6	2.8	400
	134	84	12200	7.8	4.9	700	67	42	6100	53	33	4800	31	19	2800			
	116	73	10600	6.7	4.2	600	27	17	2500	129	81	11700	8.9	5.6	800			
	111	69	10000	4.5	2.8	400	53	33	4800	134	84	12200	45	28	4100			
	111	69	10000	5.6	3.5	500	71	44	6400	85	53	7700	71	44	6400			
	122	76	11000	7.1	4.4	600	60	38	5500	107	67	9700	36	23	3300			
Ablebond 41-1	89	56	8100	13.4	8.4	1200	67	42	6100	102	64	9300	107	67	9700	4.6	2.8	400
	71	44	6400	8.9	5.6	800	89	56	8100	89	56	8100	58	36	5200			
	111	69	10000	13.4	8.4	1200	134	84	12200	142	89	12900	53	33	4800			
	134	84	12200	17.8	11	1600	111	69	10000	89	56	8100	142	89	12900			
	107	67	9700	26.7	17	2500	62	39	5700	85	53	7700	102	64	9300			
	102	64	9300	16.0	10	1500	93	58	8400	101	63	9100	93	58	8400			
Ablefilm 550	134	84	12200	8.9	5.6	800	89	56	8100	129	81	11700	53	33	4800	4.6	2.8	400
	102	64	9300	2.2	1.4	200	58	36	5200	111	69	10000	125	78	11300			
	71	44	6400	5.6	3.5	500	89	56	8100	53	33	4800	111	69	10000			
	80	50	7300	5.6	3.5	500	80	50	7300	62	39	5700	93	58	8400			
	138	86	12500	5.6	3.5	500	98	61	8800	76	48	7000	80	50	7300			
	105	66	9600	5.6	3.5	500	83	52	7500	86	54	7800	93	58	8400			

^aBased on an acceleration of 20,000 Gs

TABLE 23. COMPARISON OF BOND SHEAR STRENGTH RESULTS FOR SMALL CAPACITORS (0.127 x 0.102 x 0.102 cm)

Adhesive	Shear Strength (Newtons)															Calculated Requirement ^a		
	At 25°C			At 150°C			At 25°C After Solvent Immersion			At 25°C After 200 Temp Cycles			At 25°C After 1000 Hrs At 150°C					
	N	M/Nm ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI	N	MN/m ²	PSI
Ablebond 36-2	16	12	1700	6.7	5.2	750	14	11	1600	4.5	3.5	500	11	8.5	1200	8.2	6.3	910
	19	15	2200	12	9.2	1300	28	22	3200	6.7	5.2	750	12	9.2	1300			
	16	12	1700	6.7	5.2	750	22	17	2500	4.5	3.5	500	17	13	1900			
	21	16	2300	12	9.2	1300	31	24	3500	8.9	1.8	1000	19	15	2200			
	16	12	1700	18	14	2000	19	15	2200	17.8	1.4	2000	17	13	1900			
Average	17	13	1900	11	8.5	1200	23	18	2600	8.5	6.5	950	15	12	1700			
Epo-Tek H20E	11	8.5	1200	3.3	2.5	360	14	11	1600	16	12	1700	12	9.2	1300	8.2	6.3	910
	13	10	1500	2.2	1.7	250	21	16	2300	12	9.2	1300	45	35	5100			
	18	14	2000	3.3	2.5	360	19	15	2200	10	7.7	1100	15	12	1700			
	15	12	1700	8.9	6.8	990	13	10	1500	8.9	6.8	1000	17	13	1900			
	15	12	1700	2.2	1.7	250	14	11	1600	28	22	3200	16	12	1700			
Average	14	11	1600	4.0	3.1	450	16	12	1700	15	12	1700	21	16	2300			
Ablebond 58-6	22	17	2500	5.6	4.3	620	17	13	1900	19	15	2200	17	13	1900	8.2	6.3	910
	23	18	2600	6.7	5.2	750	14	11	1600	18	14	2000	27	21	3000			
	27	21	3000	4.4	3.4	490	26	20	2900	19	15	2200	13	10	1500			
	22	17	2500	6.7	5.2	750	22	17	2500	24	18	2600	21	16	2300			
	32	25	3600	6.7	5.2	750	20	15	2200	30	23	3300	32	25	3100			
Average	25	19	2800	6.0	4.6	670	20	15	2200	22	17	2500	22	17	2500			
Epo-Tek H44	27	21	3000	23	18	2600	32	25	3600	17	13	1900	28	22	3200	8.2	6.3	910
	--	--	---- ^b	13	10	1500	31	24	3500	23	18	2600	17	13	1900			
	30	23	3300	12	9.2	1300	45	35	5100	13	10	1500	18	14	2000			
	26	20	2900	17	13	1900	22	17	2500	11	8.5	1200	16	12	1700			
	21	16	2300	20	15	2200	27	21	3000	23	18	2600	29	22	3200			
Average	26	20	2900	17	13	1900	31	24	3500	18	14	2000	21	16	2300			

^a Based on acceleration of 20,000Gs
^b Device lost in handling

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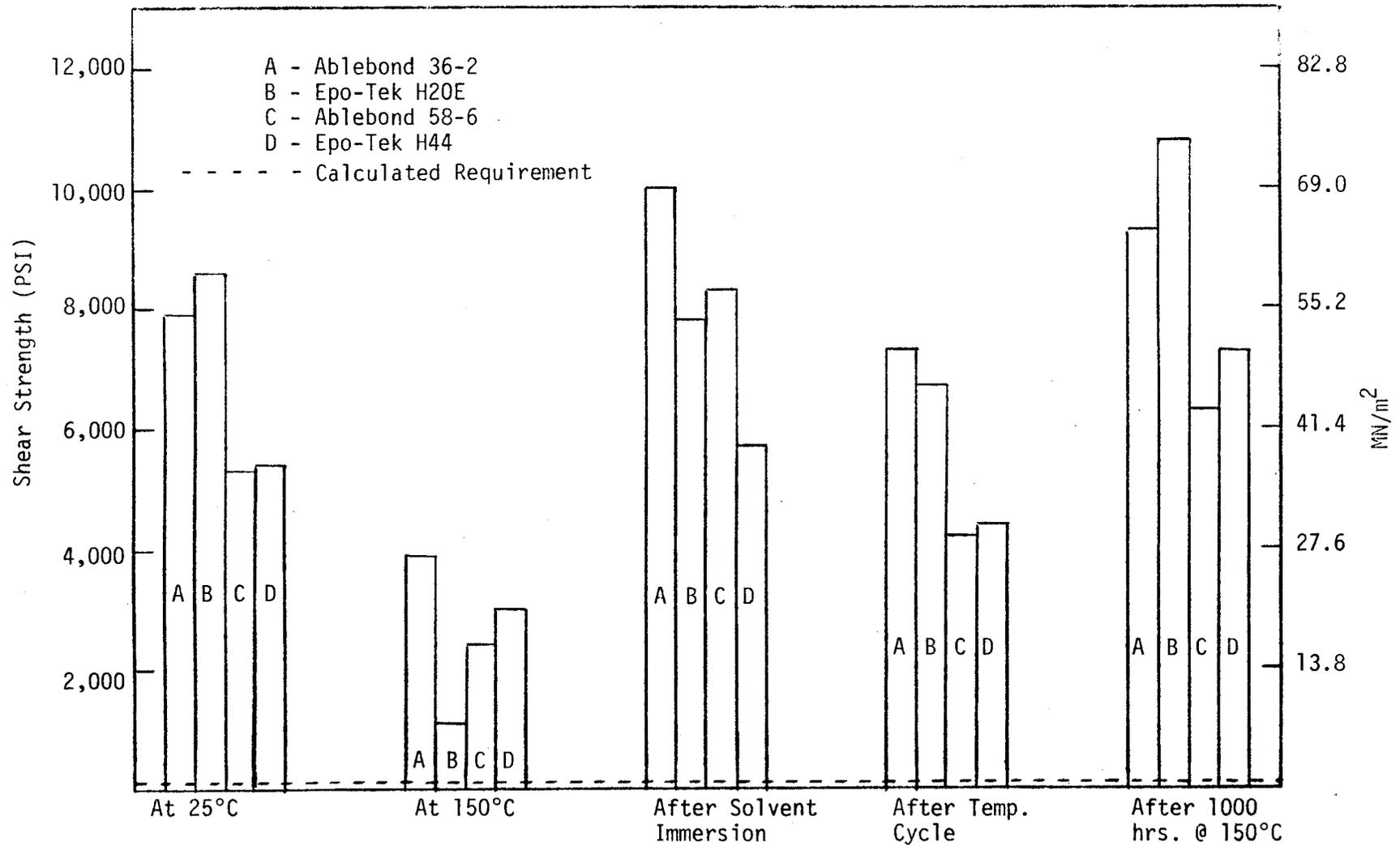


Figure 34. Comparison of Bond Shear Strength Results for Small Transistors (0.051 x 0.051 cm)

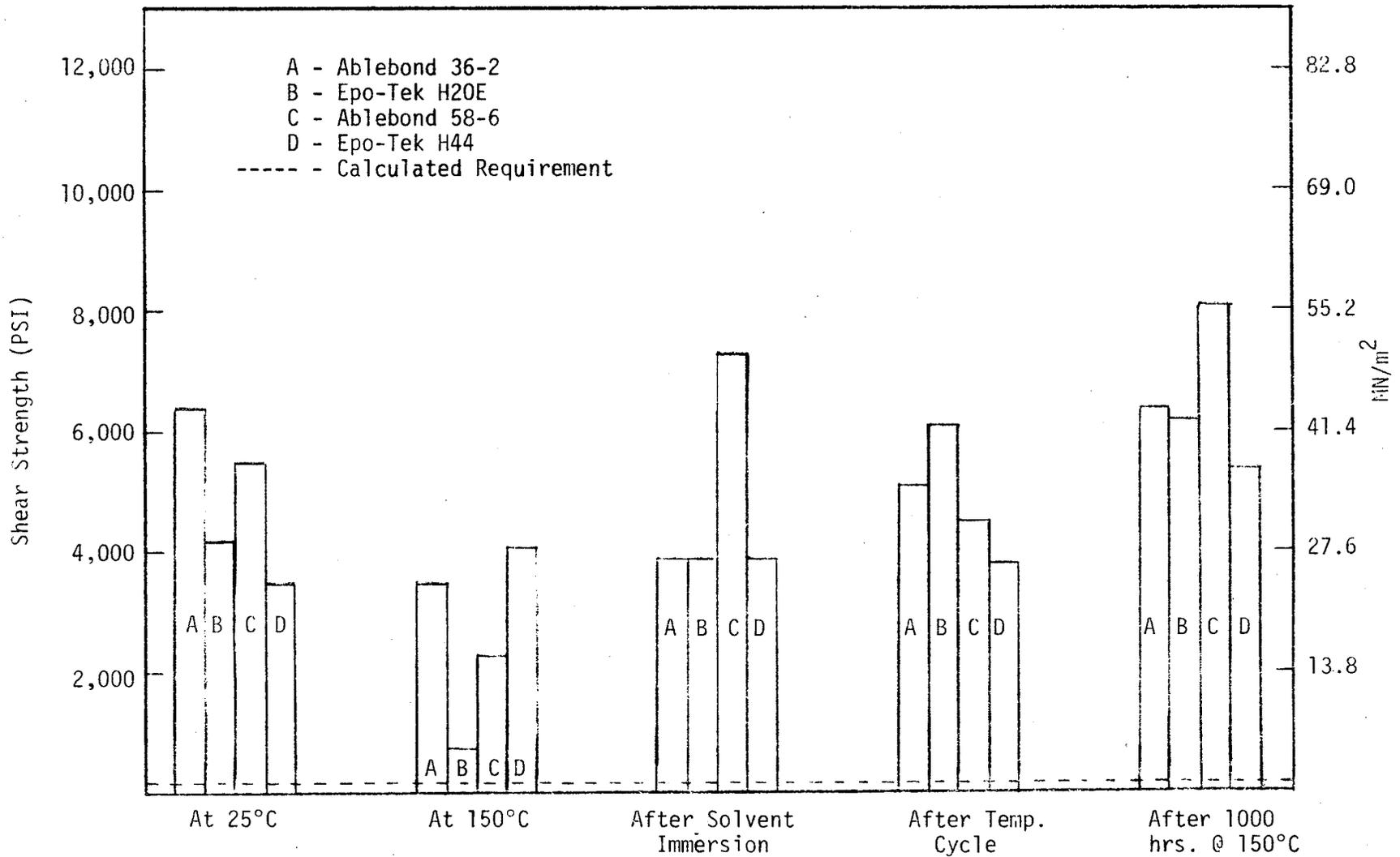


Figure 35. Comparison of Bond Shear Strength Results for Intermediate Size Transistors (0.102 x 0.102 cm)

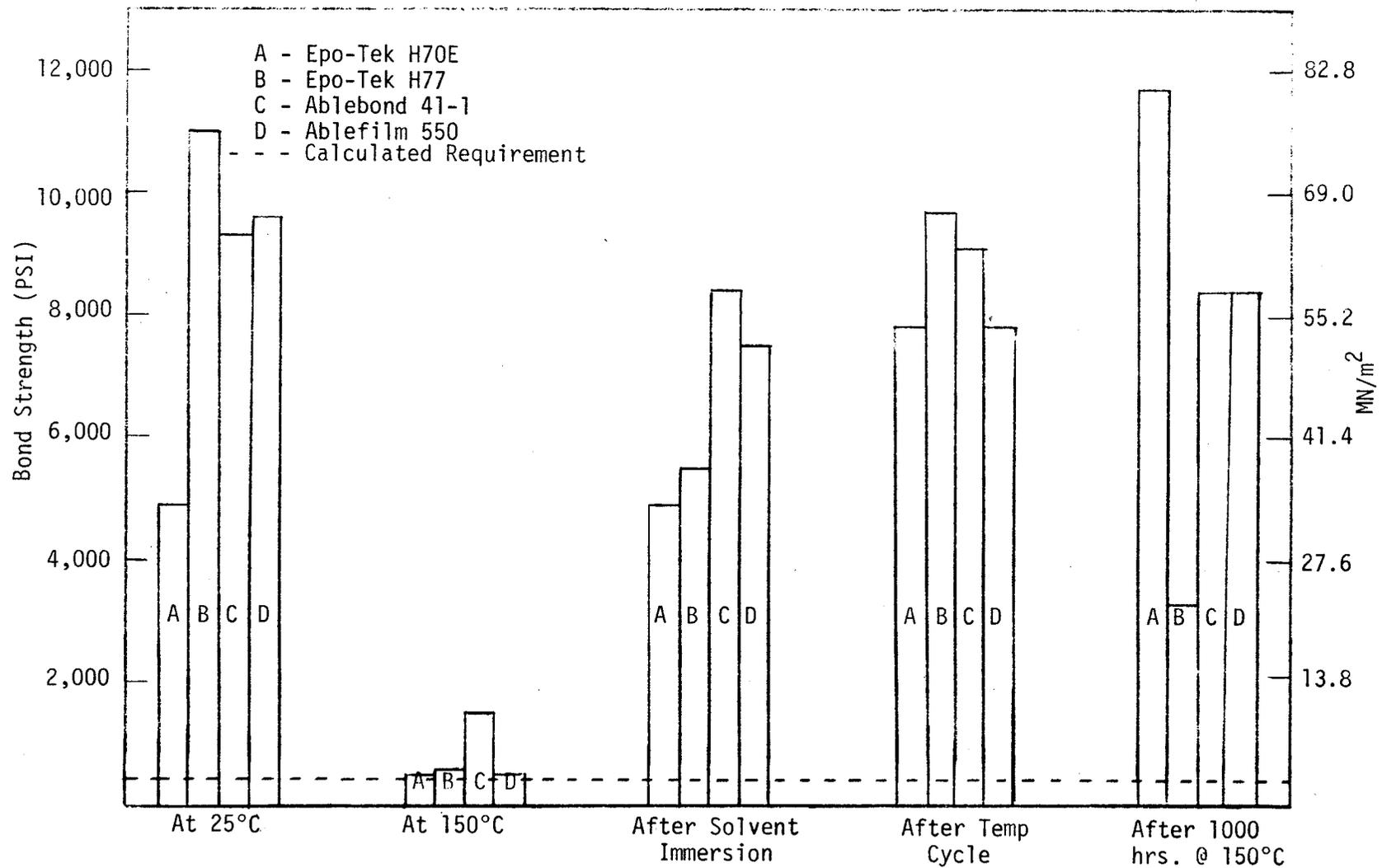


Figure 36. Comparison of Bond Shear Strength Results for Substrate Chips (0.127 x 0.127 x 0.038 cm)

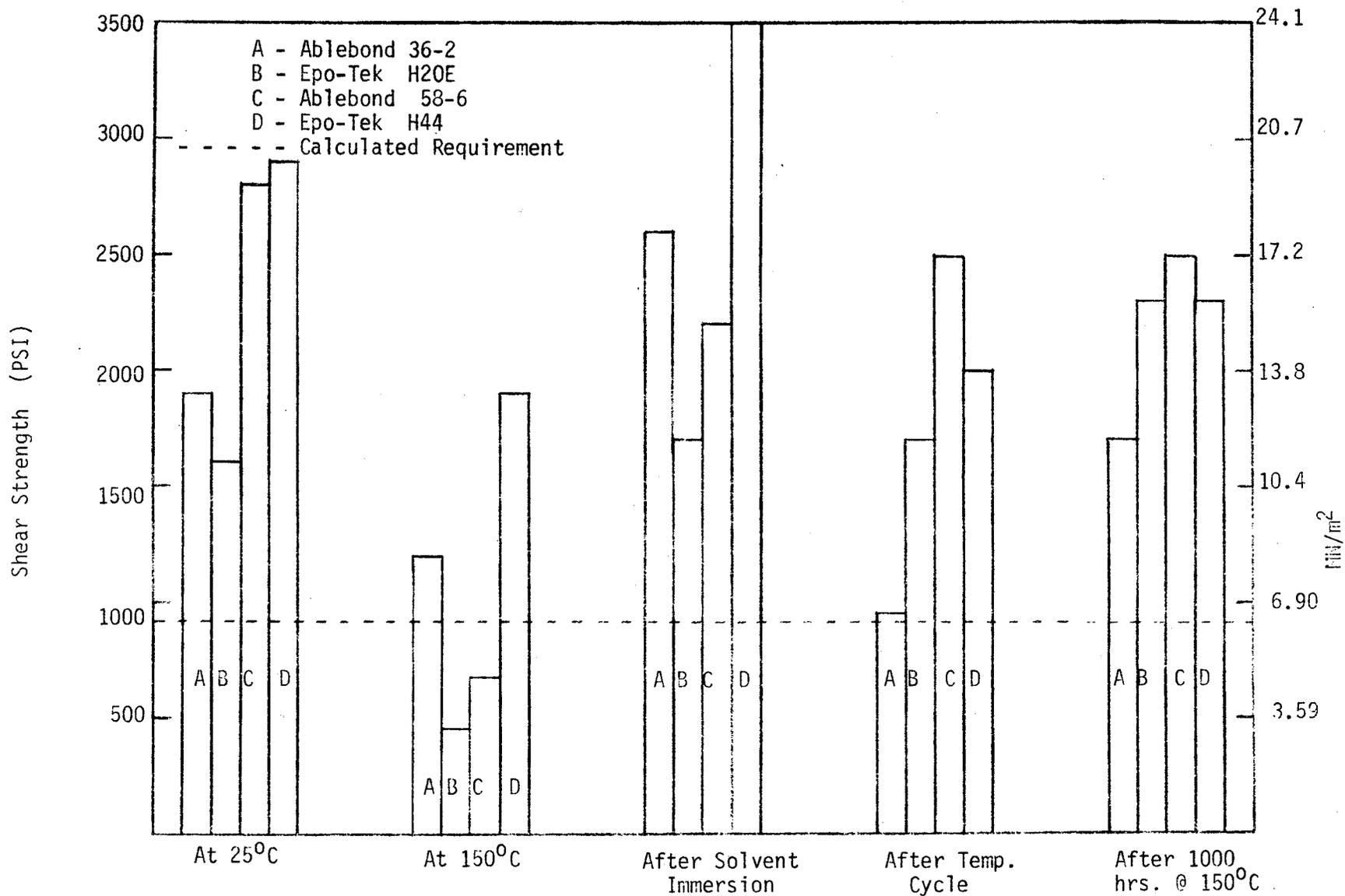


Figure 37. Comparison of Bond Shear Strength Results for Small Capacitors (0.127 x 0.102 x 0.102 cm)

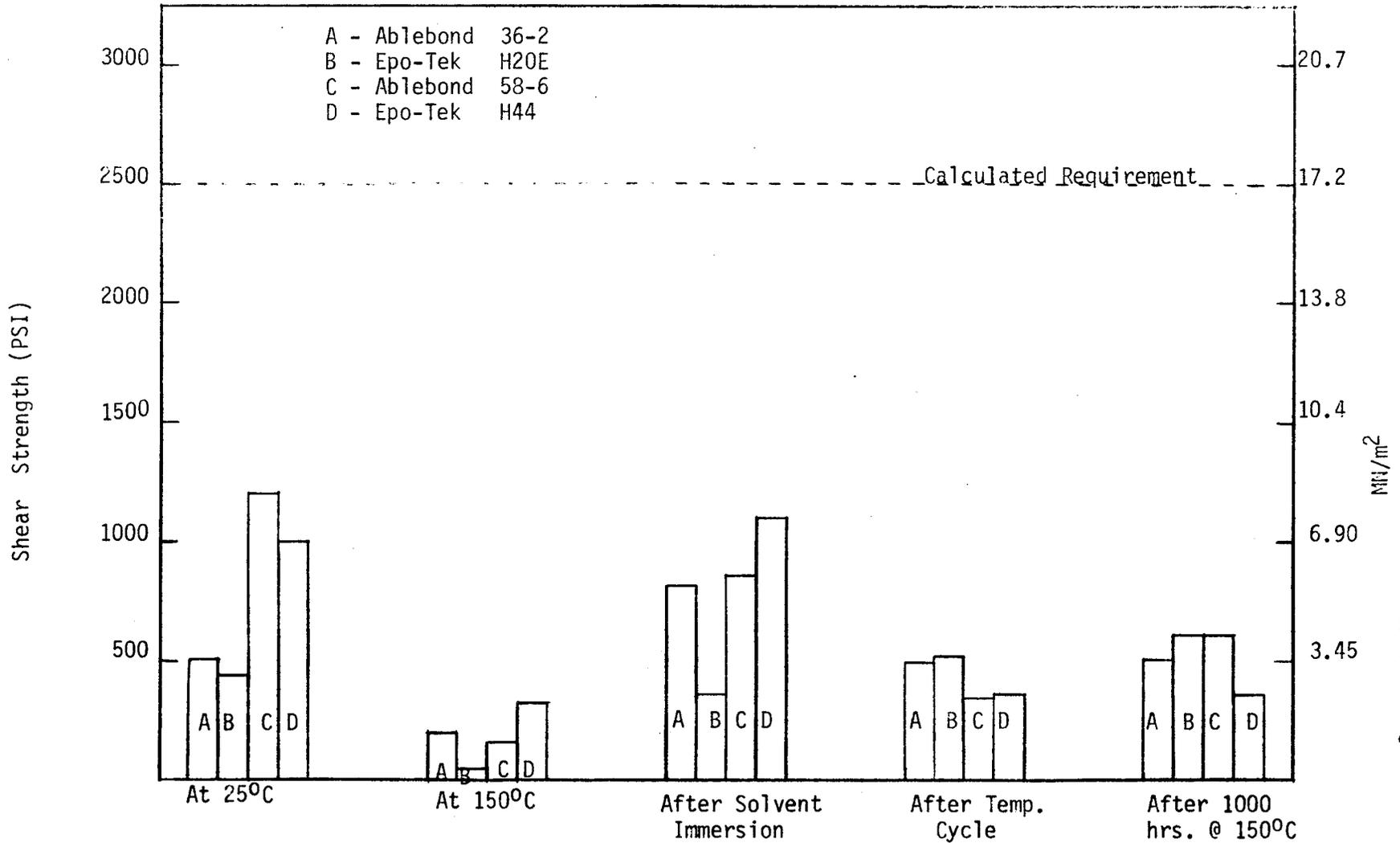


Figure 38. Comparison of Bond Shear Strength Results for Large Capacitors (0.699 x 0.216 x 0.152 cm)

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to bond the substrate chips, Table 22 and Figure 36, all of the adhesives met the calculated requirement of 2.8 meganewtons/meter² but Epo-Tek H70E and H77 and Ablefilm 550 failed to meet the minimum requirement of 6.9 meganewtons/meter² when tested at 150°C.

With the exception of Ablebond 36-2, Tables 20 and 21 and Figures 34 and 35, the electrically conductive adhesives showed little or no loss in bond strength following solvent immersion, temperature cycling, and 1000 hour storage at 150°C. (The results for Ablebond 36-2 after solvent immersion are difficult to explain since the values for the 0.051 x 0.051 cm transistors were quite high while those for the larger 0.102 x 0.102 cm transistors which should have been more resistant to solvent immersion were low. A possible explanation is that the solvents were retained longer in the adhesive under the larger transistors.) The electrically insulative adhesives (Table 22 and Figure 36) also showed little or no change with the exception of Epo-Tek H77 which showed a 50 percent loss in strength after solvent immersion and a 70 percent loss after 1000 hours at 150°C.

Based on these results, it can be seen that the equation used to calculate the bond strength requirement for semiconductors and substrates has little meaning since the adhesives greatly exceed the calculated requirement in all cases except at 150°C. The bond strength requirement will therefore be changed to require a minimum strength of 6.9×10^6 newtons/meter² (1000 psi) when measured at 25°C. In all subsequent tests the requirement will be based on the initial bond strength with the exception of the requirement at 150°C. This latter requirement will be a minimum of 3.5×10^6 newtons/meter² or 500 psi. Using the data from Tables 20, 21 and 22, the requirements to be used for semiconductors and substrates are as follows:

<u>Test or Condition</u>	<u>Semiconductors</u>	<u>Substrates</u>
At 25°C	$\geq 6.9 \text{ MN/m}^2$ (1000 psi)	$\geq 6.9 \text{ MN/m}^2$ (1000 psi)
At 150°C	$\geq 3.5 \text{ MN/m}^2$ (500 psi)	$\geq 3.5 \text{ MN/m}^2$ (500 psi)
After immersion	≥ 0.7 initial	≥ 0.7 initial
After temp. cycling	≥ 0.7 initial	≥ 0.7 initial
After 1000 hrs. at 150°C	≥ 0.8 initial	≥ 0.8 initial

In the case of the capacitors, the bond strength per unit area is difficult to calculate because the area actually bonded cannot be measured readily and varies from capacitor to capacitor. It was therefore elected to use the entire bondable area of the capacitor (whether bonded or not) in calculating the bond strength per area. The results using this method of calculation are shown in Tables 23 and 24 and Figures 37 and 38. In the case of the small capacitors, Epo-Tek H20E and Ablebond 58-6 failed to meet both the calculated requirement of 6.3 meganewtons/m² and the minimum requirements at 150°C. In the case of the large capacitors, all of the adhesives failed the calculated requirement of 17.3 meganewtons/m² (2500 psi) and while Ablebond 58-6 and Epo-Tek H44 met the minimum requirement of 6.9 meganewtons/m² in several of the tests, individual test results were scattered well above and below this value.

The amount of adhesive used for bonding the capacitors is shown in Figures 39 and 40. The small capacitors were bonded with adhesive approximating the maximum allowable. The large capacitors were bonded with approximately one-half the amount that could be used without risking shorting. (The center of the capacitor or the entire capacitor if wire bonded, could also be bonded with electrically insulative adhesive increasing the available bonding area.) It should be noted that the large capacitors are approximately 32 times heavier (Table 19) than the small capacitors while the total available bond area increased only 12 times. Selection of the proper adhesive and method of bonding is therefore very important in the case of large capacitors.

Based on the above results the requirements to be used for capacitors are as follows:

<u>Test or Condition</u>	<u>Capacitors</u>
At 25°C	≥ 10.3 x 10 ⁶ N/M ² (1500psi)
At 150°C	≥ 5.2 x 10 ⁶ N/M ² (750 psi)
At 25°C after immersion	≥ 0.7 initial
At 25°C after temp. cycling	≥ 0.7 initial
At 25°C after 1000 hrs. at 150°C	≥ 0.8 initial

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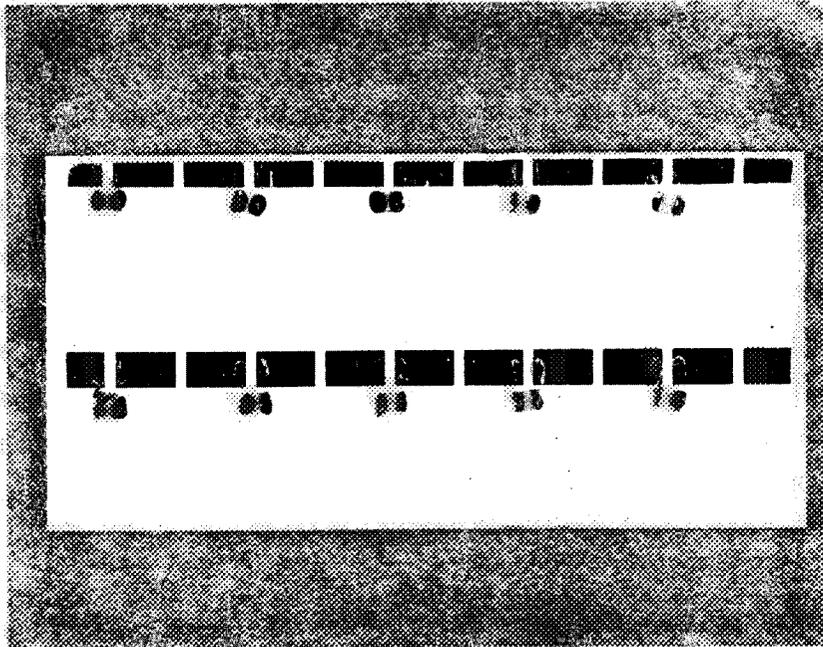


Figure 39. Small Capacitor Test Specimen Showing Bond Area Used

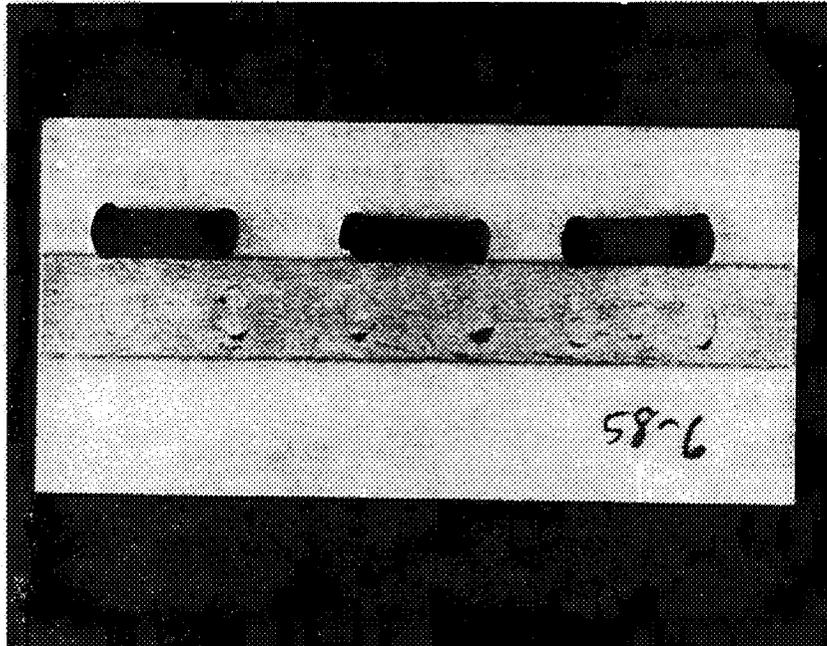


Figure 40. Large Capacitor Test Specimen Showing Bond Area Used

The requirements at 25°C and 150°C were increased over those for the semiconductors and substrates to allow for the greater weight per available bond area of the capacitors.

3.4.5 CORROSIVITY TEST RESULTS

Small dots of the paste adhesives (already mixed if a two-part adhesive) were applied to the aluminized side of a mylar film and allowed to stand for 48 hours. The film adhesive was placed on the aluminized side of the mylar film and warmed to 60 to 70°C on a hot plate causing the B-staged epoxy to melt and adhere to the film. Five samples of each adhesive were tested. After 48 hours exposure to room ambient, the adhesives (with the exception of the film adhesive) were removed from the film by washing with acetone and the film examined for corrosion as evidenced by changes in the light transmissibility. In the case of the film adhesive, the samples were examined for changes around the periphery of the adhesive. As shown in Table 25, Epo-Tek H44 was the only corrosive adhesive. It completely removed the thin film aluminum from the mylar where it was applied. The test was repeated using thin film aluminum sputter deposited on glass slides with identical results.

3.4.6 VOLUME RESISTIVITY

3.4.6.1 ELECTRICALLY CONDUCTIVE ADHESIVES

The volume resistivity test specimens for the electrically conductive adhesives were prepared using a standard 1 inch by 3 inch glass slide. The slides were cleaned with methanol and placed in a jig (Figure 41) which served as a guide for applying two strips of transparent tape 0.25 cm apart (Figure 42). Two thicknesses of tape were used to provide an adhesive layer approximately 0.008 cm thick. (It was found that the thicker layers of adhesive improved the specimen to specimen reproducibility.) A drop of the adhesive to be tested was placed between the strips and squeegeed using a single edge razor blade held at an angle of 30° to the slide to

TABLE 25. CORROSIVITY TEST RESULTS

<u>Adhesive</u>	<u>Type</u>	<u>Result</u>
Ablebond 58-6	Single component, gold filled, electrically conductive	No evidence of corrosion
Epo-Tek H44	Single component, gold filled, electrically conductive	Aluminum completely removed by corrosion under adhesive
Epo-Tek H20E	Two component, silver filled, electrically conductive	No evidence of corrosion
Ablebond 36-2	Single component, silver filled, electrically conductive	No evidence of corrosion
Epo-Tek H70E	Two component, nonconductive filler	No evidence of corrosion
Epo-Tek H77	Two component, nonconductive filler	No evidence of corrosion
Ablebond 41-1	Two component, nonconductive filler	No evidence of corrosion
Ablefilm 550	Glass supported film, nonconductive	No evidence of corrosion

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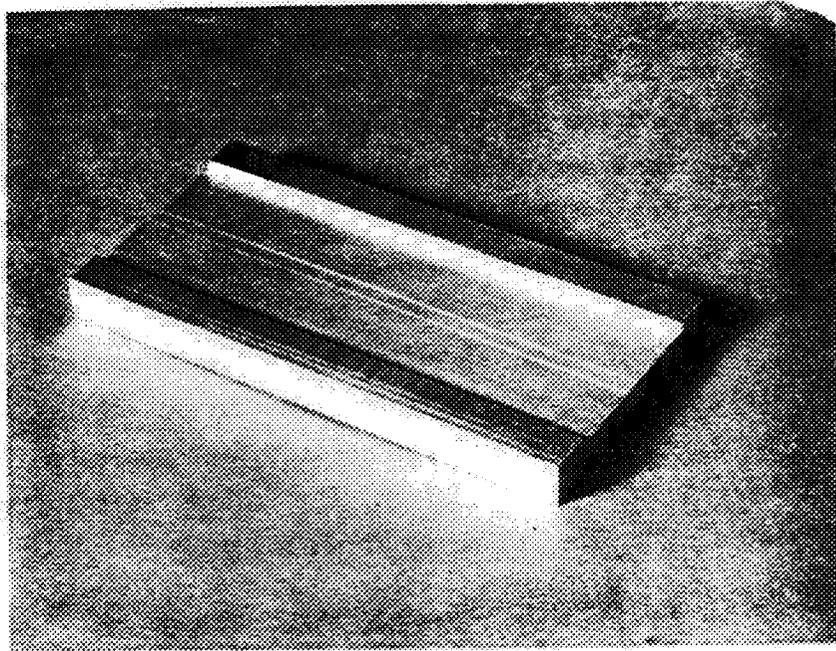


Figure 41. Jig Used to Prepare Volume Resistivity Test Specimens

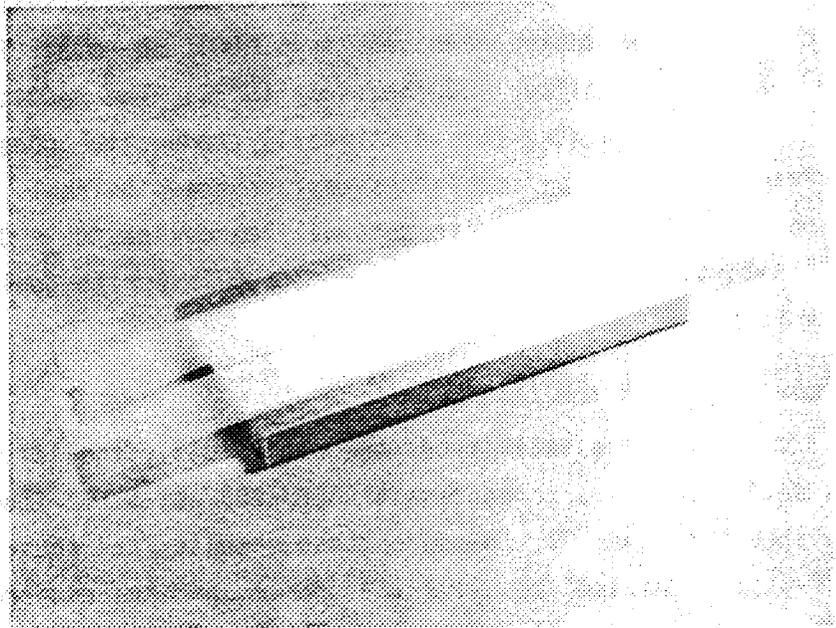


Figure 42. Jig with Transparent Tape Applied as Mask on Glass Slide

form a strip at least 5.8 cm in length. The tape was removed (Figure 43) and the adhesives were cured at 150°C for double the time recommended by the manufacturer. Resistance measurements were made using a Kiethley 179TRMS Digital Multimeter, a Hanson 6112A DC Power Supply and a General Radio 1432-M Decade Resistor in conjunction with a four-point probe system. A test fixture (Figure 44) with spring loaded contacts was used to facilitate accurate measurements. The current contacts are 5.08 cm. apart and the voltage contacts are separated from each current contact by 1.27 cm. The volume resistivity was calculated using the following formula:

$$\rho = \frac{R (wxt)}{l}$$

where ρ = volume resistivity in ohm-cm

R = measured resistance, ohms

w = width in centimeters

t = thickness in centimeters

l = length between inner pair of probes

The MSFC-SPEC-592 requirement for volume resistivity for the electrically conductive adhesives is ≤ 0.001 ohm-cm at 25°C, 60°C, 150°C and at 25°C after 1000 hours at 150°C. The results of the tests performed on the four electrically conductive adhesives (average of three specimen/adhesives) are shown in Table 26. Based on these results, the requirement will be revised to ≤ 0.0005 for the silver-filled adhesives and ≤ 0.0015 for the gold-filled adhesives.

3.4.6.2 ELECTRICALLY INSULATIVE ADHESIVES

The volume resistivity of the electrically insulative adhesives was determined in accordance with ASTM D257 at 25°C. The results are shown in Table 27. Based on these results the requirement at 25°C will be revised to $\geq 1 \times 10^{14}$ ohm-cm and the requirement at 125°C will be revised to $\geq 1 \times 10^8$ ohm-cm. These test specimens were also used for the thermal conductivity, dielectric constant, and dissipation factor measurements.

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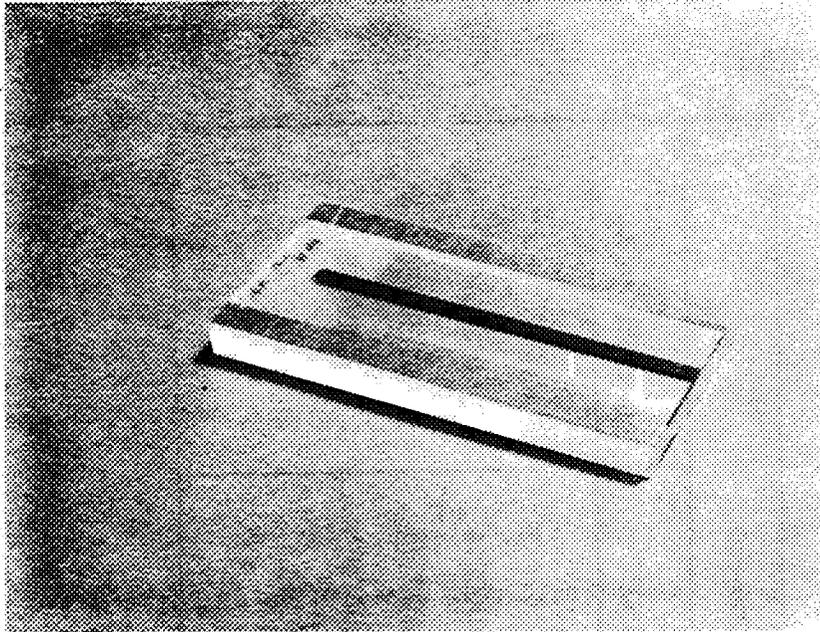


Figure 43. Jig with Test Specimen After Removal of Masking Tape

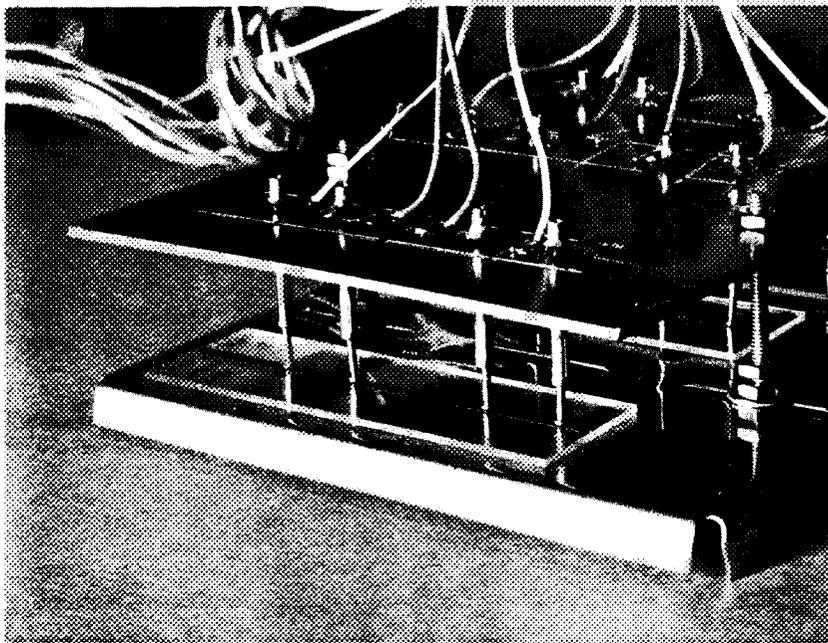


Figure 44. Test Fixture for Volume Resistivity Measurements

TABLE 26. RESULTS OF VOLUME RESISTIVITY TESTS
OF ELECTRICALLY CONDUCTIVE ADHESIVES

Adhesive	Volume Resistivity (Ohm-cm)			
	25°C	60°C	150°C	After 1000 Hours @ 150°C in N ₂
Ablebond 36-2	0.000167	0.000181	0.000214	0.000108
Epo-Tek H20E	0.000185	0.000201	0.000235	0.000150
Ablebond 58-6	0.00105	0.00115	0.00146	0.000795
Epo-Tek H44	0.000844	0.000930	0.00112	0.000722

TABLE 27. VOLUME RESISTIVITIES OF ELECTRICALLY INSULATIVE ADHESIVES

<u>Adhesive</u>	<u>Specimen Thickness (cm)</u>	<u>Specimen Density (g/cc)</u>	<u>Volume Resistivity (ohm-cm)</u>		<u>Requirement (ohm-cm)</u>
			<u>At 25°C</u>	<u>At 125°C</u>	
Ablebond 41-1	0.5428	1.68	1.50×10^{14}	2.28×10^8	$\geq 10^{12}$
	0.3117	1.70	9.60×10^{14}	3.93×10^8	$\geq 10^{12}$
79 Epo-Tek H77	0.3378	2.45	1.56×10^{14}	5.70×10^8	$\geq 10^{12}$
Epo-Tek H70E	0.4437	2.23	1.06×10^{14}	1.08×10^9	$\geq 10^{12}$
Ablefilm 550	0.2921	1.32	1.81×10^{14}	2.60×10^9	$\geq 10^{12}$

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3.4.7 THERMAL CONDUCTIVITY

The thermal conductivity measurements were performed by Geoscience Ltd., Solana Beach, California using samples prepared by Rockwell. The measurements were performed in accordance with ASTM C518 using specimens 4.40 cm in diameter at the temperatures specified in Table 28. As shown in Table 28, all of the adhesives easily met the requirements. A plot of the thermal conductivity versus sample density is shown in Figure 45. It can be seen that the thermal conductivity is proportional to the density in the case of the electrically insulative adhesives but varies in the case of the electrically conductive adhesives. (The Bakelite specimen was used as a reference standard by the test laboratory.) Based on these results the requirement will be increased to $\geq 5.0 \times 10^{-3}$ cal cm/cm² sec⁰C in the case of the electrically conductive adhesives and to $\geq 1.0 \times 10^{-3}$ cal cm/cm² sec⁰C in the case of the electrically insulative adhesives.

3.4.8 LINEAR THERMAL EXPANSION COEFFICIENT

The linear thermal expansion coefficients and glass transition temperatures of the adhesives cured in accordance with Table 9 were determined in accordance with ASTM D3381 using a DuPont Thermomechanical Analyzer (TMA). The analyses were performed at Delsen Testing Laboratories Inc., Glendale, California over the temperature range of -65 to 150°C at a heating rate of 10°C/minute in a nitrogen ambient. A repeat scan was performed on each specimen with the exception of Epo-Tek H70E as shown in Table 29. As indicated in the table, some problems were encountered in obtaining a well defined glass transition temperature in all cases. The Epo-Tek H70E also became too soft at 63°C to permit accurate measurements. The results in general indicate that the adhesives should be cured for a longer period than that used (a minimum of twice the manufacturer's recommended cure). A longer cure will increase the glass transition temperature and also change the expansion coefficients but all of the adhesives should still meet the requirements shown in Table 29.

TABLE 28. THERMAL CONDUCTIVITY TEST RESULTS

Adhesive	Specimen Thickness (cm)	Specimen Density (g/cm ³)	Measurement Temperature (°C)	Thermal Conductivity		Requirement g-cal-cm sec cm ² °C
				Btu-ft hr ft ² °F	g-cal-cm sec cm ² °C	
Ablebond 36-2	0.4186	3.48	119.4	2.05	0.00847	≥ 0.0035
Epo-Tek H20E	0.4056	3.44	120.5	1.50	0.00620	≥ 0.0035
Ablebond 58-6	0.4646	5.24	118.3	1.93	0.00797	≥ 0.0035
Epo-Tek H44	0.5070	5.51	117.2	2.28	0.00942	≥ 0.0035
Ablebond 41-1	0.5428	1.68	117.2	0.58	0.00240	≥ 0.0004
	0.3117	1.70	121.1	0.65	0.00268	≥ 0.0004
Epo-Tek H77	0.3378	2.45	121.1	1.15	0.00475	≥ 0.0004
Epo-Tek H70E	0.4437	2.23	123.9	0.90	0.00372	≥ 0.0004
Ablefilm 550	0.2921	1.32	116.5	0.33	0.00136	≥ 0.0004
Bakelite	0.4890	1.31	115.6	0.30	0.00124	--

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Rockwell International

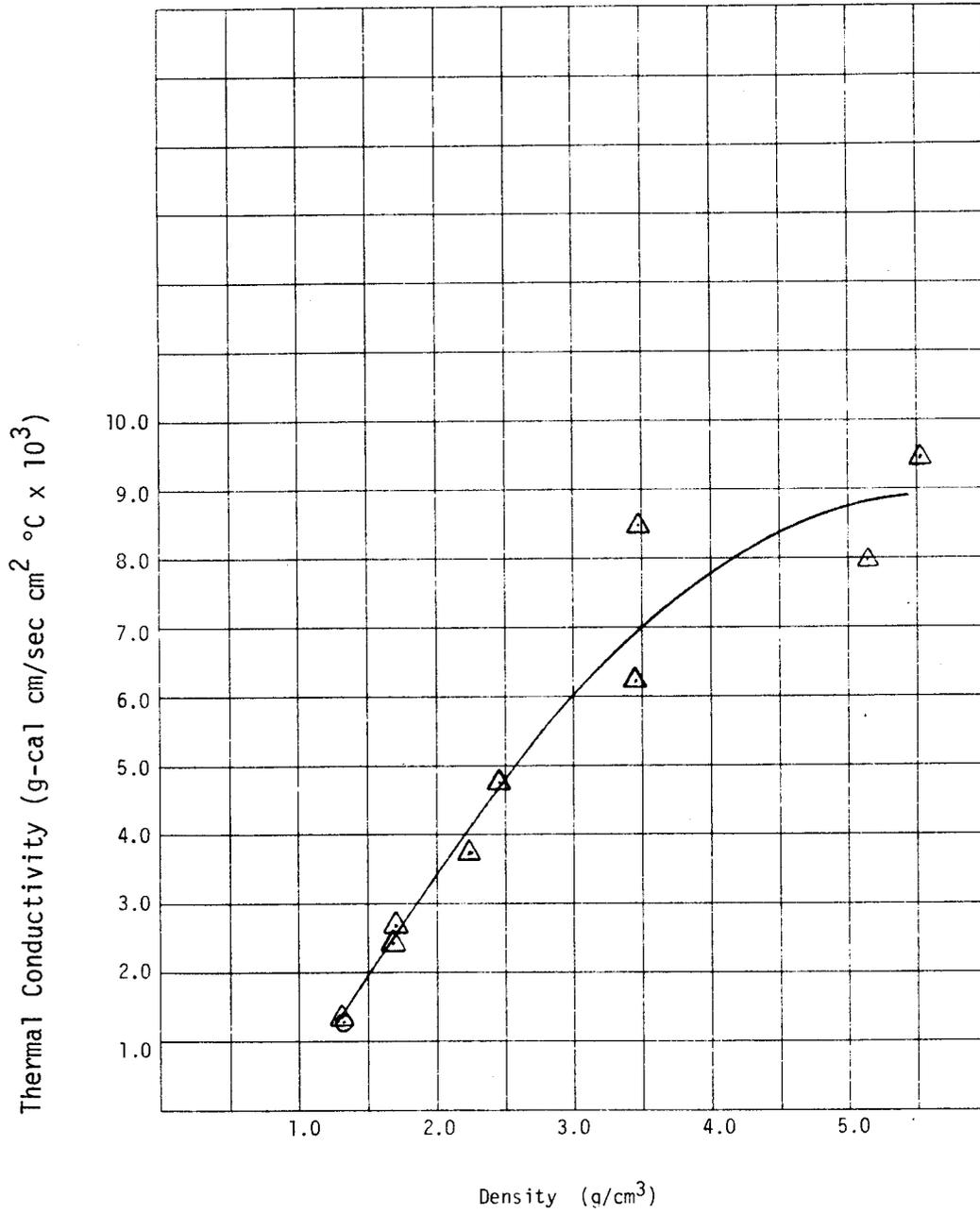


Figure 45. Plot of Thermal Conductivity Versus Density

TABLE 29. LINEAR THERMAL EXPANSION COEFFICIENTS AND GLASS TRANSITION TEMPERATURES

ADHESIVE	EXPANSION COEFFICIENT		GLASS TRANSITION (T _g °C)	REQUIREMENT
	BELOW T _g	ABOVE T _g		
Epo-Tek H20E	5.11 x 10 ⁻⁵ (to 46°C) 4.92 x 10 ⁻⁵	2.11 x 10 ⁻⁴ (above 59°C) 2.29 x 10 ⁻⁴	Undefined 36	
Ablebond 36-2	4.32 x 10 ⁻⁵ 4.47 x 10 ⁻⁵	1.81 x 10 ⁻⁴ 2.03 x 10 ⁻⁴	69 76	< 6.5 x 10 ⁻⁵ below T _g
Epo-Tek H44	3.65 x 10 ⁻⁵ (to 64°C) 3.75 x 10 ⁻⁵	1.12 x 10 ⁻⁴ (above 118°C) 1.23 x 10 ⁻⁴	Undefined 88	< 3.0 x 10 ⁻⁴ above T _g
∞ Ablebond 58-6	3.73 x 10 ⁻⁵ (to 64°C) 4.00 x 10 ⁻⁵ (to 75°C)	1.18 x 10 ⁻⁴ (above 104°C) 1.27 x 10 ⁻⁴ (above 104°C)	Undefined Undefined	
Epo-Tek H70E	3.15 x 10 ⁻⁵ (to 63°C)	--	Specimen softened at 63°C	
Epo-Tek H77	2.54 x 10 ⁻⁵ 2.46 x 10 ⁻⁵	9.74 x 10 ⁻⁵ 1.02 x 10 ⁻⁴	62 64	
Ablebond 41-1	3.04 x 10 ⁻⁵ (to 65°C) 3.20 x 10 ⁻⁵	1.03 x 10 ⁻⁴ (above 102°C) 9.61 x 10 ⁻⁵	Undefined 76	

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3.4.9 IONIC IMPURITIES

Water extract analyses were performed on the cured adhesives to determine the total ion content (electrical resistivity), hydrogen ion content (pH), chloride ion, sodium ion and potassium ion contents. Fed. Test Method Std. No. 406, Method 7071 was followed except that the adhesives were cured directly in the Erlenmeyer flasks at 150°C for double the length of time recommended by the manufacturer. The pH of the water extracts were determined using an Orion Model pH meter with a standard combination electrode. The sodium and potassium analyses were performed using atomic absorption and the chloride concentration was determined by measuring the turbidity of a sample of the water extract to which silver nitrate has been added in a Nessler tube. The results of the tests are shown in Table 30.

The ionic content in parts per million as sodium chloride was calculated using the equation:

$$\frac{23.47 (L_2 - L_1)}{W} = \text{parts per million as NaCl}$$

Where L_2 = specific conductance of sample (micromho/cm)

L_1 = specific conductance of blank (micromho/cm)

W = weight of sample in grams

23.47 = grams NaCl/mho/cm

The factor 23.47 can be obtained experimentally or calculated as follows:

$$\frac{A}{L_S} = 23.47 \text{ gms NaCl in 50 ml water/mho/cm}$$

Where: A = 1.4613×10^{-3} g NaCl in 50 ml 0.0005N NaCl

L_S = 6.225×10^{-5} mho/cm (specific conductance of 0.0005N NaCl)

The above equations assume that the samples are extracted with 50 cm³ of distilled water in accordance with Fed. Test Method STD. 406, Method 7071.

TABLE 30. RESULTS OF IONIC IMPURITY ANALYSES

Adhesive	pH	Specific Res. (ohm-cm)	Na (ppm)	K (ppm)	Cl (ppm)	Ionic Content(ppm as NaCl)
Epo-Tek H20E	4.13	37,000	32.8	29.0	24.8	1065
Ab1ebond 36-2	5.06	162,000	8.12	0.54	162.4	86
Epo-Tek H44	3.16	15,000	236.6	0.42	42.5	2930
Ab1ebond 58-6	4.50	185,000	3.23	0.00	80.0	49
Epo-Tek H70E	4.60	17,000	38.6	1.43	57.3	2560
Epo-Tek H77	4.82	109,000	17.9	2.62	147.4	227
Ab1ebond 41-1	7.04	17,000	135.8	1.20	943.0	2560
Ab1effim 550	7.83	48,000	0	1.00	291.0	775

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The specific resistivity is now used to control the total extractable ionic content of the adhesives. The ionic content as calculated above will be substituted for the specific resistivity and a maximum of 1000 ppm as NaCl (equivalent to a specific resistivity of approximately 43,000 ohm-cm) will be used as the requirement.

Based upon the results in Table 30, the overall requirements for control of the ionic content of the adhesives were selected as follows:

	<u>Recommended Requirement</u>
pH	4.0 to 9.0
Na(ppm)	≤ 50
K(ppm)	≤ 5
Cl(ppm)	≤ 300
Ionic content (ppm as NaCl)	≤ 1000

Using these requirements and examining the results in Table 30, it can be seen that the electrically conductive adhesives Ablebond 36-2 and Ablebond 58-6 are acceptable while Epo-Tek H20E is borderline based on its ionic content and Epo-Tek H44 is unacceptable based on pH, sodium content, and ionic content (Epo-Tek H44 also failed the corrosivity test). In the case of the electrically insulative adhesives, Epo-Tek H77 and Ablefilm 550 are acceptable while Epo-Tek H70E is unacceptable because of its ionic content and Ablebond 41-1 is unacceptable because of sodium, chloride and ionic contents.

3.4.10 Dielectric Constant and Dissipation Factor

The dielectric constants and dissipation factors for the electrically insulative adhesives were determined at 1KHz and 1MHz at 25°C in accordance with ASTM D150. The results of the dielectric constant measurements are given in Table 31.

TABLE 31. RESULTS OF DIELECTRIC CONSTANT MEASUREMENTS

Adhesive	Dielectric Constant		Requirement
	1 KHz	1 MHz	
Ablebond 41-1	6.06 ^a 5.91 ^b	-- 5.72	≤ 6
Epo-Tek	5.98	5.84	"
Epo-Tek H70E	5.67	5.47	"
Ablefilm 550	4.25	3.92	"

^aDensity = 1.68 g/cm³

^bDensity = 1.70 g/cm³

All of the adhesives met the dielectric constant requirement of ≤ 6. The lower density Ablebond 41-1 specimen failed with a value of 6.06. This specimen contained more voids than the higher density specimen.

The results of the dissipation factor measurements are given in Table 32.

TABLE 32. RESULTS OF DISSIPATION FACTOR MEASUREMENTS

Adhesive	Dissipation Factor		Requirement	
	1 KHz	1 MHz	1 KHz	1 MHz
Ablebond 41-1	0.0210 ^a 0.0205 ^b	-- 0.025	≤ 0.003	≤ 0.03
Epo-Tek H77	0.0051	0.013	"	"
Epo-Tek H70E	0.0061	0.019	"	"
Ablefilm 550	0.0124	0.045	"	"

^aDensity = 1.68 g/cm³

^bDensity = 1.70 g/cm³

It can be seen that none of the adhesives pass the requirement at 1 KHz of ≤ 0.003 and the Ablefilm 550 fails the ≤ 0.03 requirement at 1 MHz. Based on these results, the requirement at 1 KHz will be revised to ≤ 0.03 and the requirement at 1 MHz will be revised to ≤ 0.05.

3.5 TASK E SPECIFICATION REVISION

Based on the results of the above tests and the conclusions and recommendations derived therefrom, MSFC-SPEC-592 was revised and re-written into military specification format. The revised specification is included as Appendix A to this report.

The specification requires both qualification of the material by the supplier and qualification of the processes to be used in assembly of the hybrid microcircuit by the user. This duo qualification was believed to be essential to insure that the microcircuits will meet the reliability required for space applications.

3.6 TASK F COST DATA FOR MATERIALS TESTING

An estimate of the cost for qualification testing of electrically conductive (Type I) and electrically insulative (Type II) adhesives is given in Table 33. This estimate is based on Table V of the revised specification contained in Appendix A.

The costs include an estimate of the labor hours plus material dollars required to perform the tests. In some cases the labor hours, material dollars and independent laboratory costs were combined as in the estimated costs for testing outgassing, thermal conductivity, and coefficient of expansion. If the labor cost is assumed to be \$55 per hour, the estimated cost for full qualification of a Type I adhesive is \$12,133 and full qualification of a Type II adhesive is \$5,950. The material cost of the adhesive was not included because this cost is minor in the case of the electrically insulative adhesives and quite variable in the case of the gold and silver filled adhesives due to the fluctuation in the prices of the metal fillers.



TABLE 33. QUALIFICATION COST ESTIMATE

Test on Condition	Type I		Type II	
	Supplier	User	Supplier	User
Materials (4.5.1)	0.5 hrs	---	0.5 hrs	---
Pot Life (4.5.2)	1.0 hrs	---	1.0 hrs	---
Shelf Life (4.5.3)	29.0 hrs + \$1485	29.0 + \$150	28.0 hrs + \$1435	2.5 + \$500
Infrared Spectrum (4.5.4)	2.0 hrs	2.0 hrs	2.0 hrs	2.0 hrs
Outgassing (4.5.5.1)	\$465	---	\$465	---
(4.5.5.2)	---	\$500	---	\$500
Bond Shear Strength(4.5.6)				
Group 1 & 2	12.5 hrs + \$150	---	11.5 hrs + \$75	---
Group 3	---	12.5 hrs + \$150	---	---
Corrosivity (4.5.7)	1.0 hrs	---	1.0 hrs	---
Vol. Resist.(4.5.8.1)	14.0 hrs	2.0 hrs	---	---
(4.5.8.2)	---	---	2.0 hrs	---
Thermal Conductivity(4.5.9)	\$570	---	\$570	---
Coef. of Expansion(4.5.10)	\$250	---	\$250	---
Electrical Stability(4.5.11)	---	12 hrs	---	---
Dielectric Constant(4.5.12)	---	---	2.0 hrs	---
Dissipation Factor(4.5.13)	---	---	2.0 hrs	---
Ionic Content(4.5.14)	12.0 hrs	---	12.0 hrs	---
Sequential Test(4.5.15)	---	8 hrs + \$400	---	4.5 hrs + \$250
Total	72 hrs + \$2870	65.5 hrs + \$1700	62.0 hrs + \$2795	9.0 hrs + \$1250
Total @ \$55/hour	\$6830	\$5303	\$4205	\$1745

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4.0 CONCLUSIONS

As discussed in Paragraph 2.4, the intent of this study was to develop parametric data to establish specification requirements for the adhesives to be used in assembly of hybrid microcircuits for space applications. No attempt was made, for example, to optimize the cures from the standpoint of outgassing, or the amount of adhesive used for bonding capacitors. These parameters were found to be the ones that the adhesives most commonly failed. It is believed that the requirements as now specified, however, are essential to the reliable performance of microcircuits in space applications and that with some minor process development, the majority of the adhesives will meet the requirements.

A comparison of the performance of the adhesives with the requirements of the proposed specification contained in Appendix A is given in Table 34. It can be seen that none of the adhesives met all of the requirements. However, development of the proper cure schedules should permit the majority of the adhesives to meet the outgassing requirement and improvements in bonding techniques should permit the use of adhesives for bonding all but the largest size capacitors. Three of the adhesives failed the ionic content requirements and one was borderline while one also failed the corrosivity test. Additional processing or material changes may be required by the suppliers to meet these latter requirements.

TABLE 34. COMPARISON OF ADHESIVE PERFORMANCE WITH PROPOSED SPECIFICATION REQUIREMENTS

Specification Requirements	Adhesive							
	Ablebond 36-2	Epo-Tek H20E	Ablebond 58-6	Epo-Tek H44	Ablebond 41-1	Epo-Tek H70E	Epo-Tek H77	Ablefilm 550
Materials (3.2.1)	P	P	P	P	P	P	P	P
Pot Life (3.2.2)	"	"	"	"	"	"	"	"
Shelf Life (3.2.3)	"	"	"	"	"	"	"	"
Infrared Spectrum(3.2.4)	"	"	"	"	"	"	"	"
Outgassing (3.3.2)	"	"	"	"	"	"	"	"
Thermal Stability	"	F	"	"	F	F	"	F
Outgassed Materials	"	P	"	"	"	"	"	"
Bond Strength (3.3.3)								
Group 1	F	P	P	P	NA	NA	NA	NA
Group 2	NA	NA	NA	NA	P	P	F	P
Group 3	F	F	F	F	NA	NA	NA	NA
Corrosivity (3.3.4)	P	P	P	"	P	P	P	P
Volume Resistivity(3.3.5)	"	"	"	P	"	"	"	"
Thermal Conductivity(3.3.6)	"	"	"	"	"	"	"	"
Coefficient Expansion(3.3.7)	"	"	"	"	"	"	"	"
Electrical Stability(3.3.8)	"	"	"	"	NA	NA	NA	NA
Dielectric Constant(3.3.9)	NA	NA	NA	NA	P	P	P	P
Dissipation Factor(3.3.10)	"	"	"	"	"	"	"	"
Ionic Content (3.3.11)	P	B	P	F	F	F	"	"
Sequential Test (3.3.12)	NP	NP	NP	NP	NP	NP	NP	NP

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P = Passed F = Failed NA = Not applicable B = Borderline NP - Not performed

5.0 RECOMMENDATIONS

It is recommended that the specification prepared under this program be released as a military specification. It is also recommended that the cure schedules used for the adhesives be further evaluated to reduce the amount of potentially harmful outgassing. This outgassing may not only cause contamination and corrosion of circuit elements but may also be responsible for changes in the ohmic contact to semiconductor devices.

APPENDIX A

PROPOSED
NASA-MSFC SPECIFICATION

ADHESIVES, ORGANIC, SELECTION AND
USE IN HYBRID MICROCIRCUITS

1. SCOPE

1.1 Scope. This specification establishes the minimum requirements for screening, qualification, acceptance, and performance of organic adhesives for use in assembly of hybrid microcircuits for space applications. Adhesive procurement requires a procurement specification describing the detailed electrical, mechanical, chemical, and thermal requirements related to the specific adhesive(s) to be procured. Use of these adhesives is restricted to applications where the temperature after package sealing does not exceed 150 C.

1.2 Classification. The adhesives shall be of the following types:

Type I - Electrically conductive
Type II - Electrically insulative

2. APPLICABLE DOCUMENTS

2.1 Issues of Documents. The following documents of the issue in effect on the date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

SPECIFICATIONS

MILITARY

MIL-C-45662	Calibrating System Requirements
MIL-C-81302	Cleaning Compound Solvent, Trichlorotrifluoroethane
MIL-M-38510	Microcircuits, General Specification for
MIL-P-116	Preservation, Methods of

2.1 Issues of Documents (continued)

STANDARDS

FEDERAL

FED-STD-406 Plastics, Methods of Testing

MILITARY

MIL-STD-129 Marking for Shipment and Storage

MIL-STD-202 Test Methods for Electronics and
Electrical Component Parts

MIL-STD-883 Test Methods and Procedures for
Microelectronics

(Copies of specifications, standards, and documents required by the contractor in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other Publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on the date of invitation for bids or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING MATERIALS (ASTM)

ASTM C177 Thermal Conductivity of Materials by Means
of the Guarded Hot Plate, Test for

ASTM C518 Thermal Conductivity of Materials by Means
of the Heat Flow Meter, Test for

ASTM D150 AC Loss Characteristics and Dielectric
Constant (Permittivity) of Solid Electrical
Insulating Materials, Test for

ASTM D257 DC Resistance or Conductance of Insulating
Materials, Tests for

ASTM D3386 Coefficient of Linear Thermal Expansion of
Electrical Insulating Materials

ASTM D3850 Rapid Thermal Degradation of Solid Electrical
Insulating Materials by Thermogravimetric
Method, Test for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Adhesive Procurement Specifications. The hybrid microcircuit manufacturer shall prepare a procurement specification(s) describing the detailed electrical, mechanical, chemical, and thermal requirements related to the specific adhesives which are to be procured. The requirements shall not be less than those imposed by this specification but may be increased to reflect the specific parameters of a particular adhesive or the requirements of a particular application. The specific adhesive procurement specification(s) governing its usage shall be submitted to the cognizant contracting officer for review and approval.

3.2 Properties of Uncured Adhesive

3.2.1 Materials. The adhesive components and/or the adhesive shall be of uniform consistency, free of lumps or foreign materials, and any filler shall remain uniformly dispersed and suspended during the required pot life (3.3.2), when inspected as specified in 4.5.1. The electrically conductive fillers used in Type I adhesives shall be gold, or silver, or alloys of silver or gold or other precious metals.

3.2.2 Pot life. The minimum pot-life period shall be one hour when tested in accordance with 4.5.2. The adhesive shall be used within the manufacturer's recommended pot-life period after removal from the container, after mixing, or after thawing to room temperature in the case of premixed frozen adhesives.

3.2.3 Shelf life. The shelf life of the adhesive shall be a minimum of six months from date of receipt by the user when tested in accordance with 4.5.3. The shelf life is defined as the minimum time that the adhesive continues to meet the requirements of this test method when stored in accordance with the supplier's instructions. The shelf-life expiration date shall be affixed to the adhesive container by the supplier, and no adhesive shall be used after that date.

3.2.4 Infrared spectrum. An infrared spectrum of the uncured adhesive prepared for application, and of the individual components, if a two-component system is used, shall be obtained in accordance with 4.5.4. The infrared spectrum obtained during subsequent testing shall be identical. Any shift, disappearance, or introduction of absorption bands throughout the specified wavelength range (2.5 to 15 micrometers) of the spectrum for a subsequently procured batch of adhesive shall be cause for rejection. Minor changes in peak intensity are allowed. Changes in the infrared spectrum indicate changes in the chemical composition of the adhesive and will require requalification of the adhesive as a new material.

3.3 Properties of Cured Adhesive

3.3.1 Adhesive cure. The adhesive must be capable of meeting the requirements of this document when cured according to the supplier's instructions but not exceeding a maximum temperature of 165°C for longer than four hours. The cure schedule for the supplier tests (see 4.3 and 4.4) shall be identical for all tests and shall be reported. The cure schedule for the user tests shall be the minimum cure schedule and minimum vacuum bake specified in the user's assembly document.

3.3.2 Outgassing. Outgassing of the cured adhesive shall not exceed the values specified in Table I, determined in accordance with the test method specified therein.

TABLE I. OUTGASSING REQUIREMENTS

<u>Test</u>	<u>Requirement</u>	<u>Test Method Paragraph</u>	<u>Responsibility</u>
Thermal Stability	< 0.3% weight loss at 250 C	4.5.5.1	Vendor
Outgassed Materials		4.5.5.2	User
Moisture	< 5000 ppm		
NH ₃ and amines	Report presence		
Alcohols	Report presence		
Ketones	Report presence		
Chlorinated hydrocarbons	Report presence		

3.3.3 Bond shear strength. When tested as specified in 4.5.6, the average bond shear strength of the test specimens shall meet or exceed that specified in Table II.

3.3.4 Corrosivity. The corrosive effects of adhesives on chip metallization shall be determined in accordance with 4.5.7. There shall be no change in the light transmissibility of the aluminized Mylar in the areas where the adhesive was applied.

3.3.5 Volume resistivity. When tested as specified in 4.5.8, the volume resistivity shall meet the requirements as specified in:

Table III - Type I
Table IV - Type II

3.3.6 Thermal conductivity. When tested as specified in 4.5.9, the thermal conductivity shall meet the requirements as specified in:

Table III - Type I
Table IV - Type II

3.3.7 Coefficient of linear thermal expansion. When tested as specified in 4.5.10, the coefficient of linear thermal expansion shall meet the requirements as specified in:

Table III - Type I
Table IV - Type II

TABLE II. BOND SHEAR STRENGTH REQUIREMENTS

Bond Shear Strength Test or Condition	Requirement (Newtons/meter ²)		
	Semiconductors	Substrates	Capacitors
At 25 C	$\geq 6.9 \times 10^{6^a}$	$\geq 6.9 \times 10^6$	$\geq 10.3 \times 10^{6^c}$
At 150 C	$\geq 3.5 \times 10^6$	$\geq 3.5 \times 10^6$	$\geq 5.2 \times 10^6$
At 25 C after solvent immersion	$\geq 0.7 I^b$	$\geq 0.7 I$	$\geq 0.7 I$
At 25 C after temp cycling	$\geq 0.7 I$	$\geq 0.7 I$	$\geq 0.7 I$
At 25 C after 1000 hrs at 150 C	$\geq 0.8 I$	$\geq 0.8 I$	$\geq 0.8 I$

Note a - $\text{Newtons/m}^2 \times 1.45 \times 10^{-4} = \text{lbs/in.}^2$

Note b - I = Average initial bond strength found for the adhesive at 25 C

Note c - Calculated assuming the entire base area of the capacitor is bonded

TABLE III. GENERAL REQUIREMENTS FOR ELECTRICALLY CONDUCTIVE ADHESIVES

<u>Test or Condition</u>	<u>Units</u>	<u>Requirement</u>		<u>Test Method Paragraph</u>
		<u>Silver-filled</u>	<u>Gold-filled</u>	
Volume resistivity	ohm-cm			4.5.8.1
At 25 C		≤ 0.0005	≤ 0.0015	
At 60 C		"	"	
At 150 C		"	"	
At 25 C after 1000 hrs at 150 C		"	"	
Thermal conductivity		≥ 5.0 x 10 ⁻³		4.5.9
At 121 ± 5 C	$\frac{\text{cal cm}}{\text{cm}^2 \text{ sec}^{\circ}\text{C}}$			
Coefficient of linear thermal expansion	per deg C	Below glass transition temperature ≤ 6.5 x 10 ⁻⁵ Above glass transition temperature (if less than 150 C) ≤ 3.0 x 10 ⁻⁴		4.5.10
Electrical stability		Resistances shall not increase more than .5%		4.5.11
Ionic Impurities				
Ionic content	ppm (as NaCl)	≤ 1000		4.5.14
pH		4.0 to 9.0		
Chloride	ppm	≤ 300		
Sodium	ppm	≤ 50		
Potassium	ppm	≤ 5		

TABLE IV. GENERAL REQUIREMENTS FOR ELECTRICALLY INSULATIVE ADHESIVES

<u>Test or Condition</u>	<u>Units</u>	<u>Requirement</u>	<u>Test Method Paragraph</u>
Volume resistivity	ohm-cm		4.5.8.2
At 25 C		$\geq 1 \times 10^{14}$	
At 125 C		$\geq 1 \times 10^8$	
Thermal conductivity			
At 121 ± 5 C	$\frac{\text{cal cm}}{\text{cm}^2 \text{ sec}^\circ\text{C}}$	$\geq 1.0 \times 10^{-3}$	4.5.9
Coefficient of linear thermal expansion	deg C	Below glass transition temperature $\leq 6.5 \times 10^{-5}$ Above glass transition temperature (if less than 150 C) $\leq 3.0 \times 10^{-4}$	4.5.10
Dielectric constant			4.5.12
1 kHz		≤ 6.0	
1 MHz		< 6.0	
Dissipation factor			4.5.13
1 kHz		≤ 0.03	
1 MHz		< 0.05	
Ionic impurities			4.5.14
Ionic content	ppm (as NaCl)	≤ 1000	
pH		4.0 to 9.0	
Chloride	ppm	≤ 300	
Sodium	ppm	≤ 50	
Potassium	ppm	≤ 5	

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This requirement is limited to paste adhesives only. For film adhesives, the expansion of the film in the X and Y directions may be assumed to be equivalent to that of the glass supporting the film.

3.3.8 Electrical stability. When tested as specified in 4.5.11, the electrical stability of the Type I adhesives shall meet the requirements specified in Table III.

3.3.9 Dielectric constant. When tested as specified in 4.5.12, the dielectric constant of the Type II adhesives shall meet the requirements specified in Table IV.

3.3.10 Dissipation factor. When tested as specified in 4.5.13, the dissipation factor of the Type II adhesives shall meet the requirements specified in Table IV.

3.3.11 Ionic impurities. When tested as specified in 4.5.14, the ionic impurities shall not exceed that specified in Tables III and IV.

3.3.12 Sequential test environment. For those cases where resulting microcircuits must meet MIL-M-38510 requirements, test specimens prepared using the microelectronic adhesive in the application for which it is proposed shall withstand exposure to the test conditions specified in MIL-STD-883 as stated and modified in 4.5.14. After exposure to the complete sequence of environmental conditions, the test specimens shall show no evidence of mechanical degradation such as missing, loose or cracked capacitors, bond fillet cracking, crazing or peeling, etc. The measured bond strength shall meet the requirements at 25 C given in Table II. The measured bond resistance for the Type I adhesives shall be no greater than 0.3 ohm.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier and user are responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier and user may use their own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.1.1 Test equipment and inspection facilities. Test and measuring equipment and inspection facilities of sufficient accuracy, quality, and quantity to permit performance of the required inspection shall be established and maintained by the supplier. The establishment and maintenance of a calibration system to control the accuracy of the measuring and test equipment shall be in accordance with MIL-C-45662.

4.1.2 Inspection conditions. Unless otherwise specified herein, all inspection shall be performed in accordance with the test conditions specified in the "General Requirements" of MIL-STD-202.

4.2 Classification of inspection. The inspection requirements specified herein are classified as follows:

- a. Qualification inspection (see 4.3)
- b. Acceptance inspection (see 4.4)

4.3 Qualification inspection. Qualification inspection shall consist of all tests to determine conformance with all requirements specified herein. To insure that both the adhesive materials and the processes employing the materials are controlled, both the supplier and the user of the adhesives shall be responsible for performance of the inspection as designated in Table V.

4.3.1 Sample size. The number of samples to be subjected to each testing procedure shall be as specified in the individual test methods.

4.3.2 Failures. Failure of any of the samples to meet the testing requirements shall be cause for refusal to grant qualification approval.

4.3.3 Retention of qualification. To retain qualification, the material shall be re-evaluated at 24-month intervals.

4.4 Acceptance testing. Acceptance tests shall be performed on each lot and shall consist of the tests as specified in Table V.

4.4.1 Test lot. A test lot shall consist of all adhesive manufactured under the same batch number.

4.4.2 Sample size. The number of samples to be subjected to each testing procedure shall be as specified in the individual test methods.

4.5 Methods of examination and test

4.5.1 Materials. The adhesive components and/or system shall be examined visually at a magnification of 30x to ensure conformance with the requirements of 3.2.1.

4.5.2 Pot life. The parameters to be used in the measurement of pot life (i.e., viscosity change, skin-over, loss of bond strength, etc.) are generally material dependent. The vendor, with the concurrence of the procuring activity, shall select the procedure to be used in establishing and testing the pot life.

4.5.3 Shelf life. An unopened container of material shall be stored under the conditions specified in 3.2.3. At the end of the shelf-life period, the material shall meet the acceptance inspection requirements specified in Table V.

TABLE V. REQUIREMENTS FOR QUALIFICATION AND ACCEPTANCE TESTING

Test or Condition	Test Method Paragraph	Responsibility			
		Qualification		Acceptance	
		Supplier	User	Supplier	User
Materials (3.2.1)	4.5.1	R	O	R	R
Pot life (3.2.2)	4.5.2	R	O	R	O
Shelf life (3.2.3)	4.5.3	R	O	O	O
Infrared spectrum (3.2.4)	4.5.4	R	R	R	R
Outgassing (3.3.2)	4.5.5				
Thermal stability	4.5.5.1	R	O	R'	O
Outgassed materials	4.5.5.2	O	R	O	R'
Bond shear strength (3.3.3)					
Group 1 and 2	4.5.6	R	O	R	O
Group 3	4.5.6	O	R	O	R
Corrosivity (3.3.4)	4.5.7	R	O	R	O
Volume resistivity (3.3.5)	4.5.8	R	R (at 25 C, Type I only)	R (at 25 C, Type I only)	R (at 25 C, Type I only)
Thermal conductivity (3.3.6)	4.5.9	R	O	R'	O
Coefficient of linear thermal expansion (3.3.7)	4.5.10	R	O	R'	O
Electrical stability (3.3.8)	4.5.11	O	R	O	R'
Dielectric constant (3.3.9)	4.5.12	R	O	O	O
Dissipation factor (3.3.10)	4.5.13	R	O	O	O
Ionic content (3.3.11)	4.5.14	R	O	R'	O
Sequential test environment (3.3.12)	4.5.15	O	R	O	O

R = Required

O = Optional

R' = Skip lot frequency (2x per year or every other lot, whichever is less frequent)

4.5.4 Infrared spectrum. An infrared spectrum shall be recorded over the wavelength range of 2.5 to 15 micrometers of the uncured adhesive prepared for application or of the individual components if a two-component system is used. It will be necessary to separate the filler material from the resin in order to obtain a good spectrum. This may be accomplished by using solvents such as acetone for resin extraction or with a centrifuge. If a solvent is used, it must be desorbed or blanked out before recording the spectrum.

4.5.5 Outgassing. The vendor shall be responsible for determining the thermal stability of the adhesive when cured at the recommended time and temperature (see 3.3.1). Unless otherwise specified, this same cure time and temperature shall be used in preparing all adhesive test specimens. The user shall be responsible for determining the identities and quantities of the outgassed constituents when the adhesive is cured and subjected to vacuum bake-out in accordance with his hybrid assembly process.

4.5.5.1 Thermal stability. The thermal stability of the material shall be determined on three properly cured specimens (see 4.5.5) of approximately 10 mg each in accordance with ASTM D3850. Tests shall be performed by heating the specimens from room temperature to not less than 350 C at a heating rate not to exceed 10 C/minute in a nitrogen atmosphere with 20 ml/minute nitrogen flow.

4.5.5.2 Outgassed materials. Test specimens shall be prepared using gold- or nickel-plated Kovar packages of a configuration readily adaptable to mass spectrometric analysis. If the adhesive is to be used for substrate bonding, a substrate of proper size will be adhesively bonded in the package. If the adhesive is to be used for device bonding, dummy or actual devices sufficient to cover approximately 10 percent of the area of a substrate normally used in the package shall be bonded directly to the package base. The adhesive shall be cured using the minimum schedule and minimum vacuum bake specified in the assembly drawing. The packages shall be hermetically sealed in a dry box following vacuum bake, heated for 168 hours at 125 C, and then subjected to gas ambient analysis in accordance with MIL-STD-883, Method 1018. A minimum of three specimens shall be tested for each adhesive. The proportion (by volume) of gases such as ammonia, alcohols, amines, ketones, and other solvents shall be reported in addition to those specifically identified in Method 1018.

4.5.6 Bond shear strength. The bond shear strength of the adhesive shall be determined by using actual devices and substrate materials that will be used in production. The devices may be functional rejects. The following groups indicate the types of devices and substrates to be tested.

<u>Group</u>	<u>Device</u>	<u>Substrate</u>
1	Semiconductors with gold alloyed backing	Thin- or thick-film gold-metallized alumina or gold-plated Kovar
2	Substrate material	Gold-plated Kovar and/or nickel-plated Kovar
3	Capacitors	Thin- or thick-film gold-metallized alumina

A minimum of five semiconductor devices with alloyed gold backing (die area of 16×10^{-4} to 64×10^{-4} in.²) shall be selected for each of the Group 1 tests. For the Group 2 tests, the substrate material shall be cut into chips 0.050 inch by 0.050 inch in size, and a minimum of five chips shall be used for each test. The semiconductor chips may be bonded to either thin- or thick-film gold-metallized alumina or gold-plated Kovar substrates. Bonding of the semiconductors and substrate materials shall be accomplished using the same techniques as would be used in production except that no filleting shall be allowed for specimens tested. For the Group 3 tests, the lightest and the heaviest capacitors that will be used in production shall be selected for test. Five capacitors of each size shall be used for each test. Bonding shall be accomplished using the same techniques as would be used in production. Filleting, as allowed in production for the capacitors, is permitted. The devices in each group shall be shear tested in accordance with MIL-STD-883, Method 2019. Five devices from each group shall be tested at 25 C; at 150 C; at 25 C after consecutive immersion for one-half hour each in isopropyl alcohol, a solution of 50-volume-percent methylene chloride in trichlorotrifluoroethane, and in 100 percent trichlorotrifluoroethane; at 25 C after 100 temperature cycles in accordance with MIL-STD-883, Method 1010, Condition C; and at 25 C after 1000 hours at 150 C in a nitrogen ambient. Following solvent immersion, the specimens shall be allowed to stand for a minimum of four hours prior to shear testing. The vendor shall be responsible for the performance of the Group 1 and 2 tests, and the user shall perform the Group 3 tests.

4.5.7 Corrosivity

4.5.7.1 Paste adhesives. Smear a small sample of the uncured adhesive (already mixed if it is a two-part adhesive) on the aluminum side of a sheet of aluminized Mylar. Allow it to sit for 48 hours. Wash off the adhesive with acetone and examine the light transmissibility of the aluminized Mylar by holding the material to a light source. Any change in light transmissibility in the areas where the adhesive was smeared indicates that the adhesive has attacked the aluminum and will be grounds for failure. Five samples of each adhesive shall be tested.

NOTE: The thickness of the aluminum on the Mylar should be kept to a minimum, preferably approximately 50 to 75 angstroms. Thicker aluminum can be used, but it will be necessary to let it sit longer.

4.5.7.2 Film adhesives. For film adhesives, it will be necessary to cure a small sample of the adhesive on the aluminum side of the aluminized Mylar used above. Again, allow it to sit for 48 hours, then examine the light transmissibility of the samples. Unless the adhesive is transparent, signs of corrosion will be most apparent around the periphery of the adhesive. Any change in light transmissibility will be grounds for failure. Five samples of each adhesive shall be tested.

4.5.8 Volume resistivity

4.5.8.1 Type I electrically conductive adhesive. Test specimens shall be prepared using a standard 1-inch-x-3-inch glass slide. A jig capable of holding this slide, with two scribed lines 100 mils apart and parallel to the length, shall be the guide for applying two strips of transparent tape. Make sure there are no wrinkles or bubbles in the tape. Clean the slide with methanol and air dry. Place a drop of the adhesive to be tested between the two strips of tape. Using a single-edge razor blade, squeegee the adhesive between the tape strips, maintaining a 30-deg angle between the slide surface and the razor blade. The length of the applied strip shall be at least 2.5 inches. Carefully remove the tape and cure the adhesive according to 4.5.5. After cure, the test specimens shall be allowed to cool to room temperature. Resistance measurements shall be made using a Keithley 503 milliohm meter, or equivalent, in conjunction with a four-point probe system. A special test fixture must be made to facilitate accurate measurements. This fixture can be made of an acrylic material with four spring-loaded contacts. The contacts must be set into the acrylic so that the current contacts are two inches apart, the voltage contacts are between the two current contacts, and the voltage contacts are separated from each current contact by 0.5 inch. Place the four-point probe fixture on the strip of conductive adhesive and ensure that each of the probes is in intimate contact with the adhesive. Record the data in ohms and determine the resistivity from the following formula:

$$P = \frac{R(w \times t)}{l}$$

where P = resistivity in ohm-cm
R = measured resistance, ohms
w = width in cm = 0.25 cm
t = thickness in cm - micrometer reading of adhesive plus
glass slide minus micrometer reading of glass slide
l = length between inner pair of probes = 2.54 cm

This test shall be performed on a minimum of three specimens per test condition. Test conditions shall include measurements at 25 C, at 60 C, at 150 C, and at 25 C after 1000 hours at 150 C in a nitrogen ambient. The same specimens may be used for each test.

4.5.8.2 Type II electrically insulative adhesives. Electrically insulative adhesives shall be tested in accordance with ASTM D257 at temperatures of 25 C and 125 C.

4.5.9 Thermal conductivity. Thermal conductivity shall be tested in accordance with ASTM C177 or ASTM C518 or by use of instruments such as the Dynatech C-Matic Thermal Conductance Tester or the Colora Thermoconductometer. Measurements shall be made at 121 ± 5 C.

4.5.10 Coefficient of linear thermal expansion. The coefficient of linear thermal expansion shall be determined in accordance with ASTM D3386 over the temperature range of $-65 + 150$ C. The glass transition temperature, coefficients, and temperature ranges corresponding to different slopes of the curve shall be noted.

4.5.11 Electrical stability. The electrical stability of the electrically conductive (Type I) adhesives shall be determined by applying a current density of 139.5 amps/cm^2 (900 amps/inch^2) to bonds made with the adhesives for a period of 1000 hours at 150 C in nitrogen. Specimens shall be prepared by bonding 0.0635-cm (25-mil) square gold-plated Kovar tabs to thick- or thin-film conductor pads using the adhesive. The adhesive fillet shall be removed. A minimum of five specimens shall be prepared per adhesive. The specimens shall be series interconnected by wire bonding from the tab of one specimen to the pad of the next. The resistance of each bond shall be determined initially at 25 C prior to wire bonding by four-point probe measurement from the top of the pad using a Keithley Model 191 Digital Multimeter or equivalent. The resistance of the series of interconnected tabs shall also be determined at 25 C. The series of tabs shall be biased at a current of 0.562 ± 0.016 amps ($139.5 \pm 3.9 \text{ amps/cm}^2$ or $900 \pm 25 \text{ amps/inch}^2$) at 5.0 ± 0.3 volts for 1000 hours at 150 C. The series resistance shall be measured at approximately 200-hour intervals at 25 C out to 1000 hours. After 1000 hours, the resistance of each bond shall again be measured at 25 C.

4.5.12 Dielectric constant, Type II adhesives. The dielectric constant of the Type II adhesives shall be determined in accordance with ASTM D150 at frequencies of 1 kHz and 1 MHz at room temperature.

4.5.13 Dissipation factor, Type II adhesives. The dissipation factor of the Type II adhesives shall be determined in accordance with ASTM D150 at frequencies of 1 kHz and 1 MHz at room temperature.

4.5.14 Ionic impurities. A water-extract analysis shall be performed to determine the total ion content (electrical resistivity), hydrogen ion content (pH), chloride ion, sodium ion, and potassium ion contents. Federal Test Method Std No. 406, Method 7071, shall be followed, except that the specimens may be spread thin and cured directly in the Erlenmeyer flasks. The total ion content in parts per million as sodium chloride shall be calculated using the equation:

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$$\frac{23.47 (L_2 - L_1)}{W} = \text{parts per million as NaCl}$$

where L_2 = specific conductance of sample (micromho/cm)

L_1 = specific conductance of blank (micromho/cm)

W = weight of sample (grams)

23.47 = grams NaCl/mho/cm

The factor "23.47" may be obtained experimentally or calculated as follows, based on the use of 50 ml of water for sample extraction:

$$\frac{A}{L_s} = 23.47 \text{ gms NaCl in 50 ml water/mho/cm}$$

where $A = 1.4613 \times 10^{-3}$ gram NaCl in 50 ml 0.0005 N NaCl

$L_s = 6.225 \times 10^{-5}$ mho/cm (specific conductance of 0.0005 N NaCl)

The pH shall be read directly using an Orion Model 801 pH Meter or equivalent with a standard combination electrode by immersing the electrode in the water extract and waiting for the instrument to stabilize. Chloride ion content shall be determined by specific ion electrode (Orion or equivalent) or by comparing the turbidity of a sample of the water extract to which silver nitrate has been added in a Nessler tube to a series of chloride standards. The sodium and potassium ion contents shall be measured using atomic absorption. All results shall be stated on the basis of a one-gram sample of adhesive.

4.5.15 Sequential test environment. Suitable test specimens shall be exposed to the environmental conditions described below in the sequence given. Test specimens shall be prepared using the adhesive in the application for which it is proposed. An adequate number of test specimens shall be prepared to provide a minimum of 10 samples of each application. If several devices are of the same type but different sizes, only a representative sample (a large die and small die) need to be tested. Semiconductor die with different backing materials shall be treated as different devices. After exposure to the complete sequence of environmental conditions, the test specimen(s) shall be visually examined with the unaided eye and at 30X for any evidence of mechanical degradation such as missing, loose or cracked capacitors, bond fillet cracking, crazing or peeling, etc. Measurements shall be made of the bond strength and bond resistance (for electrically conductive adhesives).

4.5.15.1 Type I and II test conditions

1. Stabilization bake (MIL-STD-883, Method 1008, test condition C, 24 hours at 150 C).

2. Temperature cycling (MIL-STD-883, Method 1010, test condition C, except 200 cycles).

3. Constant acceleration (MIL-STD-883, Method 2001, test condition D, Y₁ orientation only).

4. High-temperature storage (240 hours at 125 C followed by 72 hours at 150 C).

5. PREPARATION FOR DELIVERY

5.1 Preservation, packaging and packing. Unless otherwise specified by the procuring activity, the preservation, packaging and packing shall be in accordance with MIL-P-116, Method III.

5.2 Marking. Each container shall be marked in accordance with MIL-STD-129. Marking shall include, but not be limited to, the following information:

- a. Manufacturer's name and location
- b. Material trade name
- c. Net weight or volume
- d. Lot number, batch number and date of manufacture
- e. Storage limitations and shelf life expiration date
- f. Number and revision letter of this specification
- g. Number and revision letter of procurement specification

6. NOTES

6.1 Ordering data. Procurement documents should specify, but not be limited to, the following information:

- a. Title, number and revision letter of this specification
- b. Title, number and revision letter of procurement specification
- c. Size and number of containers required
- d. Manufacturer's product designation
- e. Request for test data

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